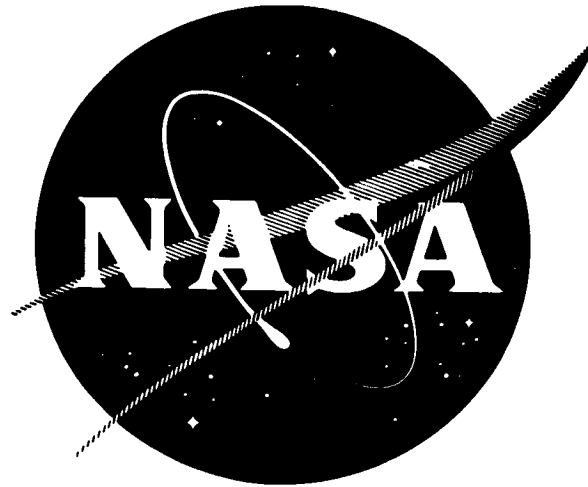


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# **EVALUATION OF HIGH STRENGTH COLUMBIUM ALLOYS FOR ALKALI METAL CONTAINMENT**

**INTERIM REPORT NO. 2**

**Covering the Period  
July 25, 1962 to July 10, 1964**

by  
**L.B. ENGEL JR.  
R.G. FRANK**

prepared for  
**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION**  
**CONTRACT NAS 3-2140**

**SPACE POWER AND PROPULSION SECTION  
MISSILE AND SPACE DIVISION**

**GENERAL  ELECTRIC**  
**CINCINNATI, OHIO 45215**

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J. W. Semmel, Jr., Manager  
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## FOREWORD

The work described herein was performed by the General Electric Company under the sponsorship of the National Aeronautics and Space Administration under Contract NAS 3-2140. Its purpose, as outlined in the contract, is to evaluate the corrosion resistance of high strength columbium alloys to boiling and condensing potassium for possible use as containment materials in space electric power conversion systems.

This work was administered for the General Electric Company by R. G. Frank, Manager, Physical Metallurgy, Materials and Processes. The experimental investigations were performed by L. B. Engel, Jr., R. G. Carlson, and D. N. Miketta with the assistance of W. H. Hendrixson and H. J. Bauer.

Mr. R. L. Davies of the National Aeronautics and Space Administration was the Technical Manager for this study. Recognition is also given to Mr. T. A. Moss for his assistance in monitoring the program.

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## I. INTRODUCTION

## I. INTRODUCTION

Advanced turboelectric space power systems will require the containment of alkali metals at temperatures approaching 2200°F, with possible hot spots at considerably higher temperatures. Only refractory alloys can be considered for the construction of such a system and, even so, critical components of the system will be strength limited. Consequently, there will be a reduction in system weight when the strongest alloy possible is used within the bounds of system reliability. Considering the strength-weight requirement for service at temperatures up to 2200°F in conjunction with the complex fabrication and joining involved in the construction of the system, it is apparent that columbium alloys are most promising for satisfying these requirements in the near future.

In addition, it is imperative to provide materials with suitable corrosion resistance to alkali metals and, although not fully documented, columbium alloys have shown very promising results in this respect. However, the only extensive experience that has been obtained in the containment of alkali metals by columbium alloys has been with the relatively weak Cb-1Zr alloy, and it is generally recognized that a stronger alloy would offer a significant advantage for the advanced space electric power systems.

This investigation was undertaken to document the behavior of AS-55 and D-43 columbium base alloys in an environment of refluxing potassium for periods of time up to 10,000 hours at temperatures on the order of 2000°F.

The AS-55 designation represents a columbium-base alloy with about 5%W, 1%Zr, and 0.06%C which is arc melted with an addition of about 1%Y to the electrode, much of the yttrium being removed as the volatile YO during vacuum melting. The primary reason for the addition of yttrium to the alloy is to enhance both the fabricability by the removal of oxygen during melting and the weldability by gettering of oxygen during welding. The D-43 designation represents a columbium-base alloy with about 10%W, 1%Zr and 0.1%C. Inclusion of both alloys in the program permits the evaluation of two levels of carbon in the same alloy system and provides two alloys with varying strength/fabricability/weldability characteristics; the AS-55 alloy having a higher degree of fabricability and weldability and the D-43 alloy a higher strength potential<sup>1</sup>. Also, both alloys contain reactive elements (Zr, Y) that are strong oxide formers which react with the oxygen in solution in the columbium-tungsten base. Alloys of this type are expected to be compatible with alkali metals.

Both alloys are essentially in the Cb-W-Zr-C quaternary alloy system and their superior strength is achieved by a combination of two basic strengthening mechanisms: solid solution of tungsten in a columbium matrix and dispersion of carbides formed by reactions between zirconium, columbium and carbon. The use of the latter strengthening mechanism to increase the temperature capabilities of refractory alloys is generally considered to be very effective. However, the carbides that are formed during the processing of the material and that subsequently inhibit slip by impeding dislocation glide must be chemically and thermally stable in the presence of potassium for a period of at least 10,000 hours in the temperature range of 2000° to 2200°F. Therefore, determining the chemical and thermal stability of the complex carbides in AS-55 and D-43 alloys under the above conditions forms the major technical objective of this program.

## II. SUMMARY

## II. SUMMARY

Corrosion testing of AS-55, Cb-1Zr and D-43 alloy reflux capsules containing purified potassium was conducted at 2000°F for 5,000 hours in an ultra high vacuum chamber capable of achieving a vacuum of  $2 \times 10^{-10}$  torr. A vacuum in the  $10^{-8}$  torr range was maintained throughout the test. The condensing rate of the potassium contained within the 1-inch diameter x 11-inch long capsules was approximately  $37 \pm 12$  lbs/hr/ft<sup>2</sup> at 2000°F.

Evaluation of two capsules, AS-55 alloy (#8) and Cb-1Zr alloy (#6), after the 5,000-hour exposure revealed only a very slight amount of general corrosion. This was evidenced by some solutioning and the presence of dark stains visible at the liquid-vapor interface and other locations.

However, an improved transfer system was used in filling the second AS-55 alloy (#12) and the D-43 alloy (#2) capsules with potassium and no signs of staining were observed in either capsule after the 5,000-hour exposure, as was observed in the two earlier capsules. Also, the improved transfer system provided the first reasonable agreement of oxygen analysis between the mercury amalgamation techniques (33 to 50 ppm) and the zirconium-getter method (22 ppm). A very small amount of white, non-metallic deposit, that is believed to be Y<sub>2</sub>O<sub>3</sub>, was observed in the AS-55 alloy capsule (#12). No attack of grain boundaries in either the weldments or recrystallized sheet was observed in any of the alloys.

Although some coalescence of the carbides appears to have occurred in the AS-55 alloy, stress-rupture testing of specimens machined from the capsule wall after the 5,000-hour, 2000°F test exposure indicates no loss in strength as a result of the exposure to potassium. Chemical analyses of samples obtained from the capsule walls revealed no mass transfer of carbon in either the AS-55 or D-43 alloys. However, the results of the chemical analysis of all three alloys provides an indication that oxygen is leached from material high in oxygen by the potassium condensate.

Similar capsule testing for a time period of 10,000 hours has been conducted on AS-55, Cb-1Zr and D-43 alloys and the materials are now being evaluated. No visual signs of gross corrosion are apparent.

### III. TECHNICAL PROGRAM

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#### A. Materials Procurement

The AS-55 alloy sheet used to manufacture capsules for this program was produced by the General Electric Company. Three heats were utilized in the capsule preparation, i.e., NAS-555, NAS-5514 and NAS-5515. Heats NAS-555 and NAS-5514 were consumably arc cast, forged, warm rolled and subsequently cold rolled to produce 0.080-inch thick sheet; heat NAS-5515 was consumably arc cast, extruded, warm rolled and then cold rolled to 0.082-inch thick sheet.

The D-43 alloy sheet was procured from the E.I. duPont de Nemours Company where it was produced from an ingot which was consumably arc cast, extruded, warm rolled and finished by cold rolling to 0.080-inch thick sheet.

The Cb-1Zr alloy sheet was purchased from the Stellite Division of Union Carbide and Carbon Corporation. The ingot was electron beam melted, forged and cold rolled to 0.100-inch thick sheet.

Summaries of the processing details of the previously mentioned heats are given in Appendix A. Chemical analyses of the finished sheets are presented in Table I and the final heat treating history is shown in Table II.

#### B. Capsule Preparation

Eight capsules, two of Cb-1Zr alloy, two of D-43 alloy and four of AS-55 alloy, were fabricated from the 0.080-inch thick sheet which was described in Section III A of this report. The sheets were roll formed into cylinders 11 inches long and approximately 1 inch in diameter. A set of formed capsules together with machined end caps and bend specimens are shown in Figure 1. The heat treatment histories of the capsules before forming, after forming and after TIG welding are shown in Table II.

The TIG welding was performed in a chamber which was first evacuated to a pressure of less than  $1 \times 10^{-4}$  torr and then back filled with helium which had been passed through a dry-ice trap. Prior to welding each capsule, a weld pass was made on a sheet of titanium to getter oxygen in the system and thereby minimize contamination of the weldments during the



TABLE I

## CHEMICAL ANALYSES OF AS-55, Cb-1Zr AND D-43 ALLOY SHEETS

## USED IN THE FABRICATION OF REFLUX CORROSION CAPSULES

<u>Alloy</u>	<u>Heat</u>	<u>Condition</u>	Chemical Analyses, w/o					<u>N(2)</u>	<u>H(2)</u>
			<u>W</u>	<u>Zr</u>	<u>Y</u>	<u>C(I)</u>	<u>O(2)</u>		
AS-55	NAS-555(3)	Stress-Relieved 2300°F/1 Hour	5.35	0.81	0.73	0.059	0.0187	0.0143	---
AS-55	NAS-5514(3)	Stress-Relieved 2300°F/1 Hour	5.25	0.78	0.02	0.058	0.0463	0.0226	0.0003
AS-55	NAS-5515(4)	Stress-Relieved 2200°F/1 Hour	5.17	0.86	0.27	0.064	0.0210	0.0205	0.0003
Cb-1Zr	Stellite- 519	Recrystallized 2200°F/1 Hour	--	1.29	--	0.004	0.0144	0.0028	0.0005
D-43	DuPont-322	Stress-Relieved 2200°F/1 Hour	9.6	0.99	--	0.085	0.0041	0.0030	0.0001

-----

- (1) Average of Two Analyses; Complete Cross Section of Sheet Used for Sample.
- (2) Average of Two Vacuum Fusion Analyses; Complete Cross Section of Sheet Used for Sample.
- (3) Analyses of Metallic Elements Performed on Samples Taken from As-Rolled Plate.
- (4) Analyses of Metallic Elements Performed on Samples Taken from Ingot.

TABLE II

THERMAL HISTORY OF REFLUX CORROSION CAPSULESPRIOR TO FILLING WITH POTASSIUM

Capsule No.	Alloy	Ingot No.	Heat Treatment, °F(1)		
			<u>Prior To Forming</u>	<u>After Forming</u>	<u>After TIC Welding</u>
6 & 7	Cb-1Zr	Stellite - 519	2200	2300	2200
8	AS-55	NAS-5514	2300	2800	2400
11 & 12	AS-55	NAS-5515	2200	2800	2400
9	AS-55	NAS-555	2300	2800	2400
2 & 4	D-43	D-43-322	2200	None	2400

-----

(1) All Heat Treatments Performed for One Hour in a Vacuum of  $<1 \times 10^{-4}$  Torr.

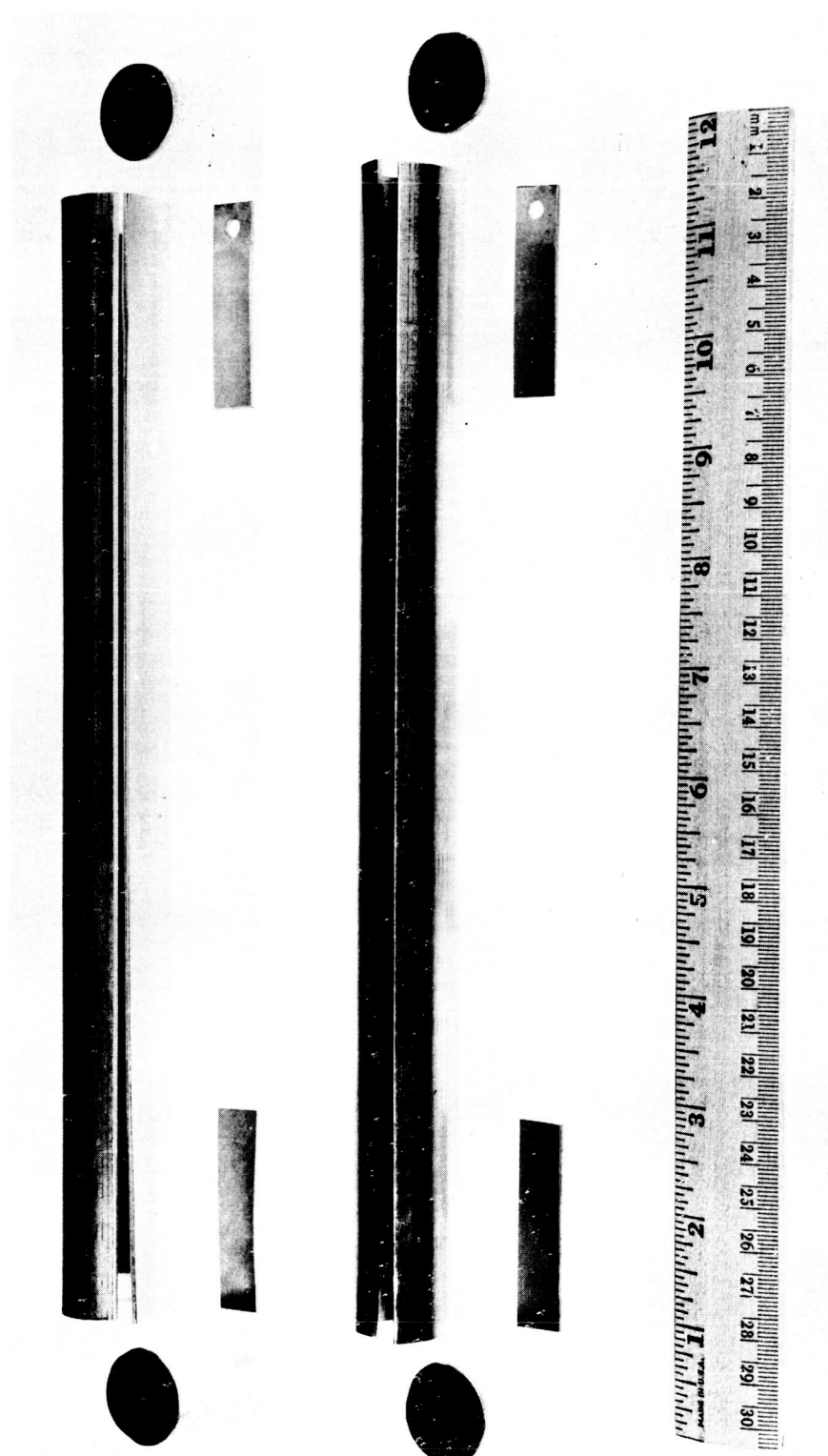


Figure 1. Typical Reflux Corrosion Capsules, End Caps, and Bend Specimens Prior to Assembly and Welding; Top: AS-55 Alloy (C63032016), Bottom: D-43 Alloy (C63032017).

welding process. The filler material used during the welding was the same as the capsule material. Table III shows typical chemical analyses of welds obtained in this manner in Cb-1Zr alloy sheet. Typical welded capsule tubes are shown in Figure 2. Although weldments made in the manner described above are sound and are considered satisfactory for the intended application, significant improvements have been made in TIG welding equipment and procedures for refractory alloys in the past several years, as evidenced by work conducted under recent NASA sponsored research and development contracts<sup>2-4</sup>.

Two fill tubes, 0.5-inch OD x 0.040-inch thick wall, were fabricated from each of the AS-55 and Cb-1Zr alloy capsule materials and TIG welded to the top end caps which also were fabricated from the capsule material. Subsequently, the top end cap and fill tube subassemblies and the bottom end caps were TIG welded to the formed and welded capsule cylinders. In the case of the two AS-55 alloy capsules #8 and #9, short lengths of 0.500-inch OD x 0.040-inch thick wall, centerless ground Cb-1Zr alloy tubing were TIG welded to the top of the AS-55 alloy fill tube to facilitate crimping of the fill tube after filling with potassium. This length of Cb-1Zr alloy tubing was removed after the capsule was sealed. The capsule components were pickled in an acid solution of 20%HF+20%HNO<sub>3</sub>+60%H<sub>2</sub>O, rinsed in water and given a final cleaning with ethyl alcohol prior to specimen placement and assembly welding. A schematic of the AS-55 alloy capsule design is shown in Figure 3. The Cb-1Zr alloy capsule design (capsules #6 and #7) is the same as that shown for the AS-55 alloy capsule with the exception that it is monometallic.

All four capsules (#6, #7, #8 and #9) contained 0.080-inch thick x 0.5-inch wide x 2-inch long bend specimens in the liquid and vapor regions as shown in Figure 3. These bend specimens were made from the same material as their respective capsules. The interiors of the capsules, with bend specimens in position, were examined with a borescope and no detrimental defects could be observed in the welds. The capsules were leak checked with a helium mass spectrometer as prescribed in MIL-Std-271B and found to be leak tight. A radiographic examination of the four capsules showed their welds to be sound. A photograph of the Cb-1Zr alloy capsules #6 and #7 and the AS-55 alloy capsules #8 and #9 prior to filling with potassium is shown in Figure 4.

Capsules #6, #7, #8 and #9 were charged with slagged, filtered, distilled and hot trapped potassium as received from Mine Safety and Appliance Research Corporation using the facilities shown in Figure 5 and the procedures outlined on the following page:

TABLE III

## CHEMICAL ANALYSES OF Cb-1Zr ALLOY WELDS

<u>Test</u>	Sheet Thickness <u>Inch</u>	<u>Sample Location</u>	<u>Chemical Analyses, ppm</u>				<u>Remarks</u>
			<u>C</u>	<u>O</u>	<u>N</u>	<u>H</u>	
1	0.040	Weld	-	390	145	1	Chamber vacuum purged once to $1 \times 10^{-4}$ torr prior to filling with He which is passed through a dry-ice cold trap
		Parent Metal	-	360	160	2	
2	0.060	Weld	-	126	27	4	
		Parent Metal	-	115	45	5	
3	0.025	Weld	80	406	78	-	
		Parent Metal	58	382	84	-	

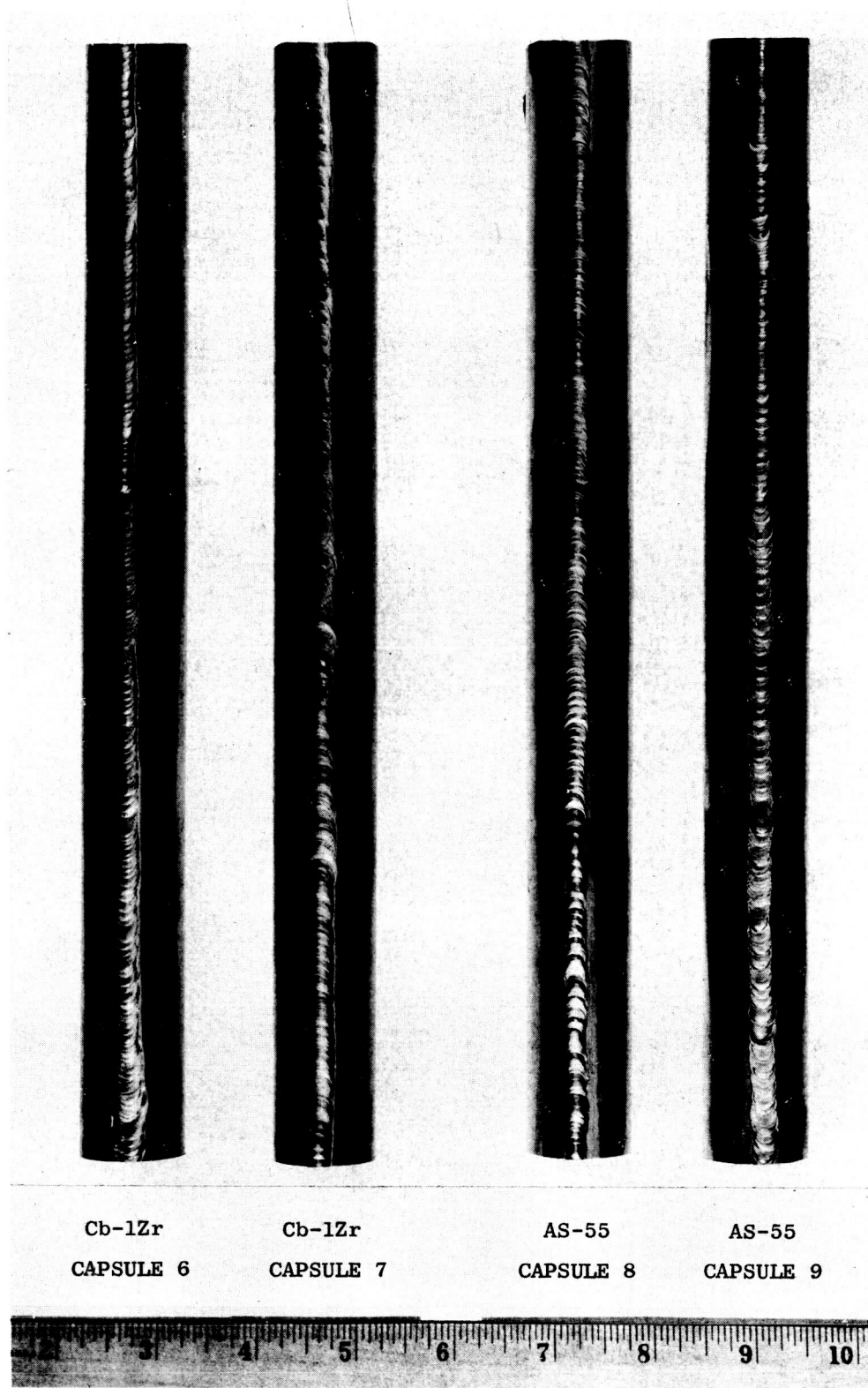


Figure 2. Formed and TIG Welded Reflux Corrosion Capsules of AS-55 and Cb-1Zr Alloys. (C2060126)

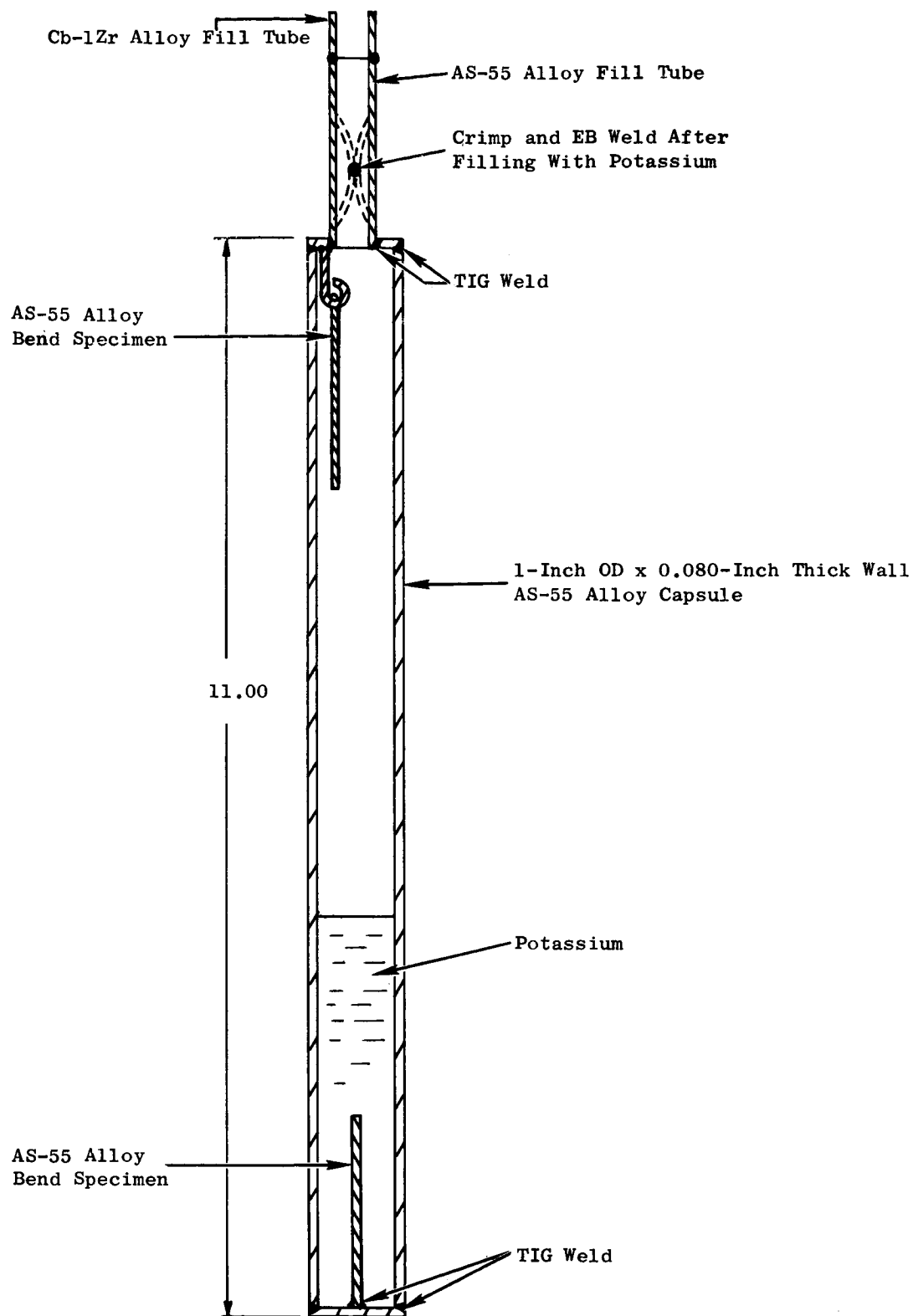


Figure 3. AS-55 Alloy Reflux Corrosion Capsule #8 Showing Fill Tube and Location of Bend Specimens.



Figure 4. AS-55 Alloy Capsules #8 and #9 (Left) and Cb-12r Alloy Capsules #6 and #7 (Right) Prior to Filling with Potassium.  
(C2050119)



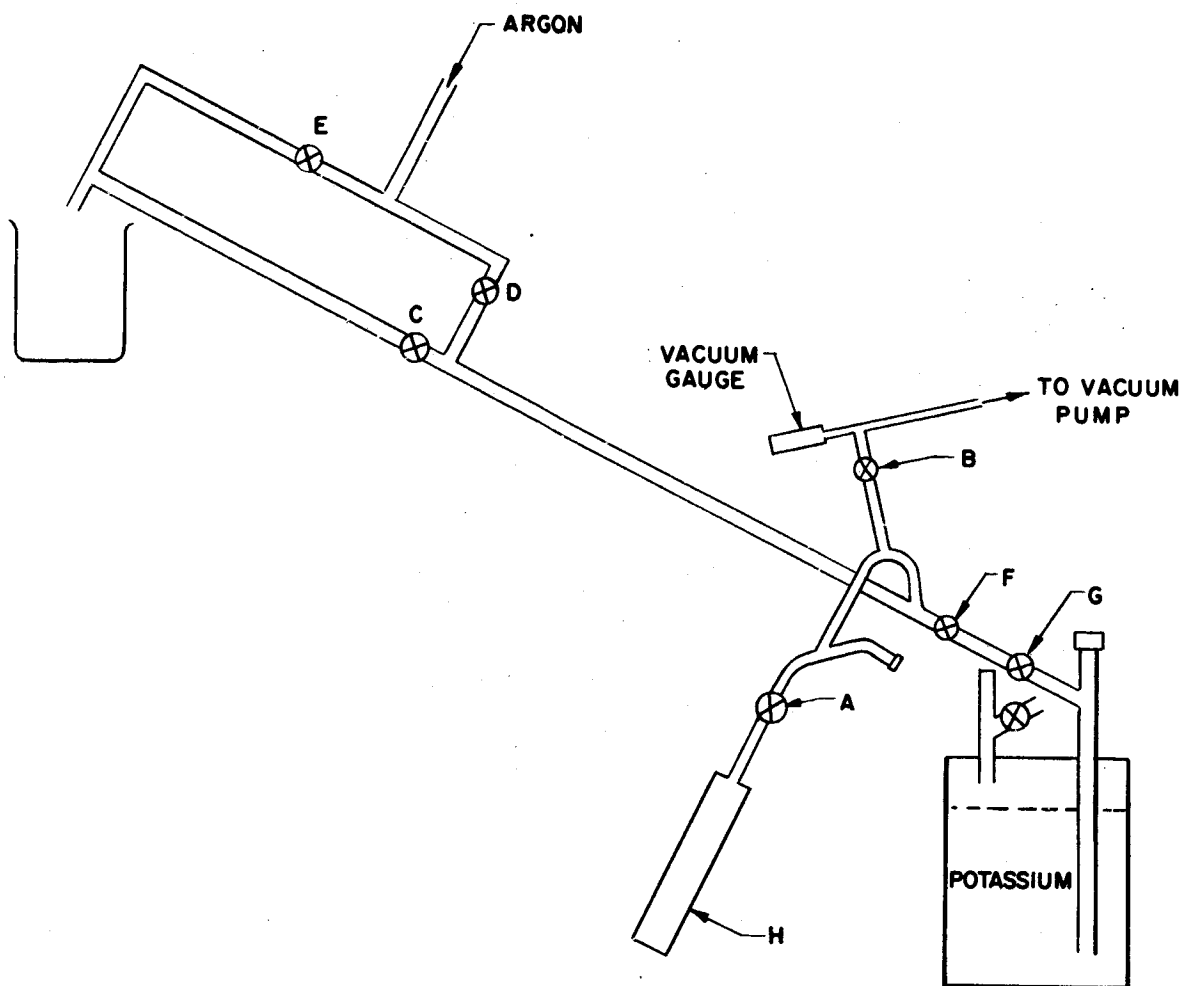


Figure 5. Apparatus for Filling Capsules #6, #7, #8 and #9 with Potassium.

1. Evacuate the capsule loading system to below  $1 \times 10^{-3}$  torr.
2. Heat the system to above 150°F to permit outgassing.
3. Close the vacuum valve (A) to seal the capsule under a vacuum, and close vacuum valve (B) to isolate the system from the vacuum pumps.
4. Flush the system, other than the capsule, with argon by opening valves (C and D).
5. Re-evacuate the system to below  $1 \times 10^{-3}$  torr by closing valves (C and D) and opening valve (B).
6. Refill the system with argon.
7. Close one argon valve (D) and leave the other argon curtain flow valve (E) open.
8. Force potassium through the system by opening valve (F) and the potassium storage container valve (G) until potassium flows out of the argon curtain opening.
9. Allow the potassium to stay in the system for 3 to 5 minutes.
10. Force new potassium into the system as in 8.
11. Close the valve (C) to seal potassium in a sampling tube, and close valve (F) to confine a measured amount of potassium.
12. Open the capsule valve (A) and simultaneously open the argon valve (D) to force the measured length of potassium into the capsule (H).
13. Seal the capsule by closing valve (A), pinching down the tubing and welding in a vacuum by electron beam.

The chemical analysis of the slagged, filtered, distilled and hot trapped potassium used to fill capsules #6, #7, #8 and #9 as obtained from the vendor is listed in Table IV. The potassium was analyzed for metallic elements by the Nuclear Materials and Equipment Corporation using spectrographic techniques and for oxygen by MSA Research Corporation using the mercury amalgamation method. The potassium was sampled at the same time and under the same conditions that the four reflux capsules, #6, #7, #8 and #9, were filled and was analyzed for

## CHEMICAL ANALYSES OF POTASSIUM

Sample Identification	Date Analyzed	Chemical Analyses <sup>(1)</sup> , ppm																								Remarks
		O	Fe	S	Co	Mn	Al	Mg	Sn	Cu	Pb	Cr	Si	Ti	Mo	V	Be	Ag	Nd	Ba	Ca	Sr	Zr	Ni		
<u>Shipping Container No. A-4</u>																										
Vendor analysis	2-1-62	<100 <sup>(2)</sup>	50	<1	<10	3	1	20	2	<1	1	1	100	<5	<2	<5	<5	<1	75	2	15	1				Si and Ca levels are high due to contamination from glass sample tube.
<u>Capsule Filling<sup>(5)</sup> - Capsule No's. 6, 7, 8 and 9</u>																										
Sample cast in capsule direct from container	12-5-62	760 <sup>(3)</sup>																								Sample taken directly in evacuated Co-I2r capsule at time of filling of capsule No's. 6, 7, 8 and 9.
Sample obtained from transfer line	12-5-62	7 <sup>(2)</sup>																								} Sample taken in evacuated and valved stainless steel pipe after filling capsule No's. 6, 7, 8 and 9.
Sample obtained from transfer line	12-5-62	16 <sup>(2)</sup>																								
<u>Shipping Container No. 137</u>																										
Vendor analysis	1-2-63	17 <sup>(2)</sup>	<5	<10	<5	<1	5	4	<5	<1	<5	<5	40	<5	<3	<1	<1	<1	45					<10	<5	} Sample obtained in stainless steel pipe open on one end to flowing argon and capped in air.
GE analysis	1-23-63	141 <sup>(2)</sup>	-----																							
GE analysis	1-28-63	190 <sup>(2)</sup>																								
GE analysis	1-28-63	200 <sup>(2)</sup>																								
GE analysis	1-28-63	219 <sup>(2)</sup>	-----																							
GE analysis	2-6-63	426 <sup>(3)</sup>																								Sample obtained in stainless steel pipe open on one end to flowing argon, capped in air, transferred to Co-I2r capsule in argon and sealed under vacuum.
GE analysis	2-6-63	406 <sup>(3)</sup>																								Sample obtained in valved stainless steel pipe, transferred to Co-I2r capsule in argon and sealed under vacuum.
GE analysis	10-15-64	53.7 <sup>(2)</sup>	-----																							} Sample obtained in evacuated stainless steel pipe and capped in air.
GE analysis	10-15-64	52.4 <sup>(2)</sup>																								
GE analysis	10-16-64	44.3 <sup>(2)</sup>																								
GE analysis	10-16-64	50.8 <sup>(2)</sup>	----																							
General Atomics	10-5-64	83.5 <sup>(4)</sup>																								} Sample obtained in evacuated stainless steel pipe, capped in air and transferred to Cu capsules in purified helium.
General Atomics	10-5-64	73.1 <sup>(4)</sup>																								
NUMEC	3-10-63	--	60	<10	<5	3	3	15	<5	3	<5	20	65	<5	<3	<1	<1	<1	100					<10	15	
<u>After Hot Trapping<sup>(6)</sup></u>																										
200 hours at 1300°F	3-11-63	20.8 <sup>(2)</sup>	25	<10	<5	<1	5	9	<5	3	<5	5	50	<5	<3	<1	<1	<1	100					<10	5	Sample obtained in stainless steel pipe open on one end to flowing argon and capped in air.
<u>Capsule Filling<sup>(7)</sup> - Capsule No's. 11, 12, 2 and 4</u>																										
Sample cast inside tank	4-15-63	33 <sup>(2)</sup>																								
Sample cast inside tank	4-15-63	36 <sup>(2)</sup>																								
Sample cast inside tank	4-15-63	33 <sup>(2)</sup>																								
Sample cast inside tank	4-15-63	22 <sup>(3)</sup>																								
Sample obtained from transfer line	4-15-63	39 <sup>(2)</sup>																								
Sample obtained from transfer line	4-15-63	53 <sup>(2)</sup>																								

- (1) All Metallics Determined From KCl by Spectrographic Techniques by the Nuclear Materials and Equipment Corporation.
- (2) Oxygen as  $K_2O$  was Determined by the Mercury Amalgamation Method.
- (3) Oxygen was Determined by Zirconium-Gettering Technique, Related Data Reported in Appendix B.
- (4) Oxygen was Determined by Neutron Activation at General Atomics.
- (5) Potassium From Shipping Container No. A-4.
- (6) Potassium From Shipping Container No. 137.
- (7) Potassium From Shipping Container No. 137 After Hot Trapping for 200 Hours at  $1300^\circ F$ .

oxygen by the zirconium-gettering technique. In this technique a 0.005-inch thick corrugated zirconium strip of known chemistry, with a total surface area of 20 square inches, was inserted in a 1-inch OD x 0.080-inch thick wall x 6-inch long Cb-1Zr alloy capsule. The capsule was filled with 7.14 grams of potassium using the facility illustrated in Figure 5, sealed under vacuum by electron beam welding techniques and heated for 100 hours at 1400°F in a vacuum of  $10^{-5}$  torr. After exposure, the zirconium strip was removed from the capsule and analyzed for oxygen, nitrogen, hydrogen and carbon. From the interstitial analyses, and on the assumptions that the interstitial elements completely reacted with the zirconium and no gaseous pickup occurred during the transfer of the potassium, the oxygen and carbon contents of the potassium were calculated to be 760 ppm (an average of four analyses) and 133 ppm, respectively. The data are presented in Table IV and Table I of Appendix B. No significant change in the nitrogen or hydrogen could be detected in the zirconium getter material.

Oxygen analyses of the potassium used to fill capsules #6, #7, #8 and #9 also were obtained by the mercury amalgamation method at General Electric. However, in this case the samples were drawn at a later date. The samples were taken by removing sections of tubing between valves G and F of the filling apparatus shown in Figure 5. Analysis of these samples produced values of 7 and 16 ppm oxygen as  $K_2O$ , Table IV. These data indicated that contamination was occurring during the filling of the capsules. To overcome this problem, a new potassium transfer facility was successfully developed, which subsequently was used in the filling of the remaining two AS-55 alloy capsules (#11 and #12) and the two D-43 alloy capsules (#2 and #4).

The potassium transfer facility that was developed is similar to the transfer facility at NASA Lewis Research Center and consists of a 30 KV, electron beam welding chamber, shown in Figure 6, which incorporates facilities for transferring potassium directly from the hot trap to the capsules in a vacuum of  $5 \times 10^{-5}$  torr with a leak rate of 1.4 micron-cubic feet per hour. The pumping system consists of a 10-inch oil diffusion pump and a 80 cfm Stokes Microvac mechanical forepump. The vacuum is measured with a National Research Corporation Type 501 ionization gauge and Model 710B control circuit. Figure 7 shows an external view of the facility with the hot trap in place; Figure 8 shows the general capsule arrangement inside the chamber. The potassium is transferred to the capsules by pressurizing the hot trap, filling the stainless steel ladle with the potassium to the required level (30 grams) and then pouring the potassium through the stainless steel funnel into the capsule

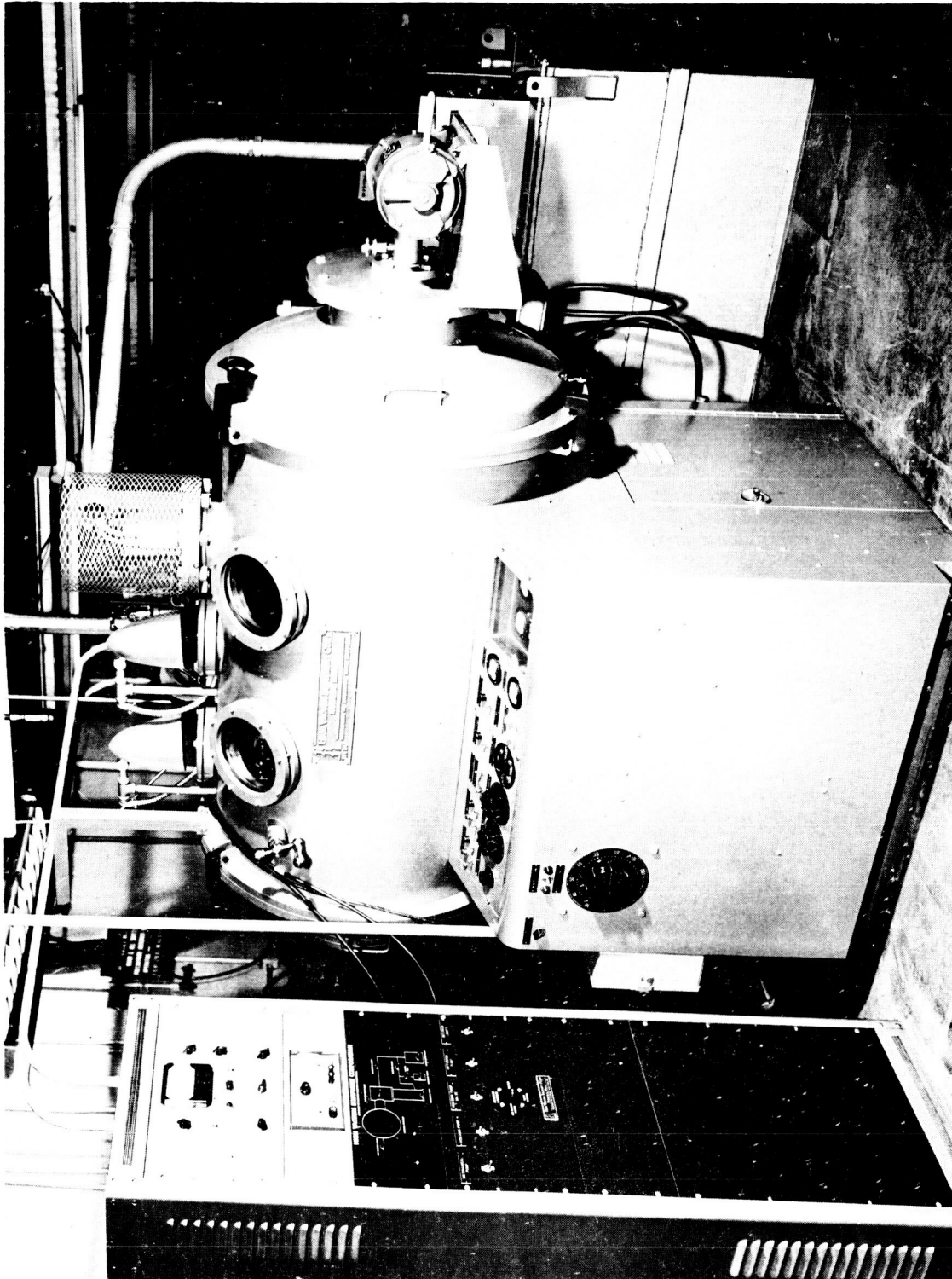


Figure 6. 30 KV Electron Beam Welding Facility. (C1022014)

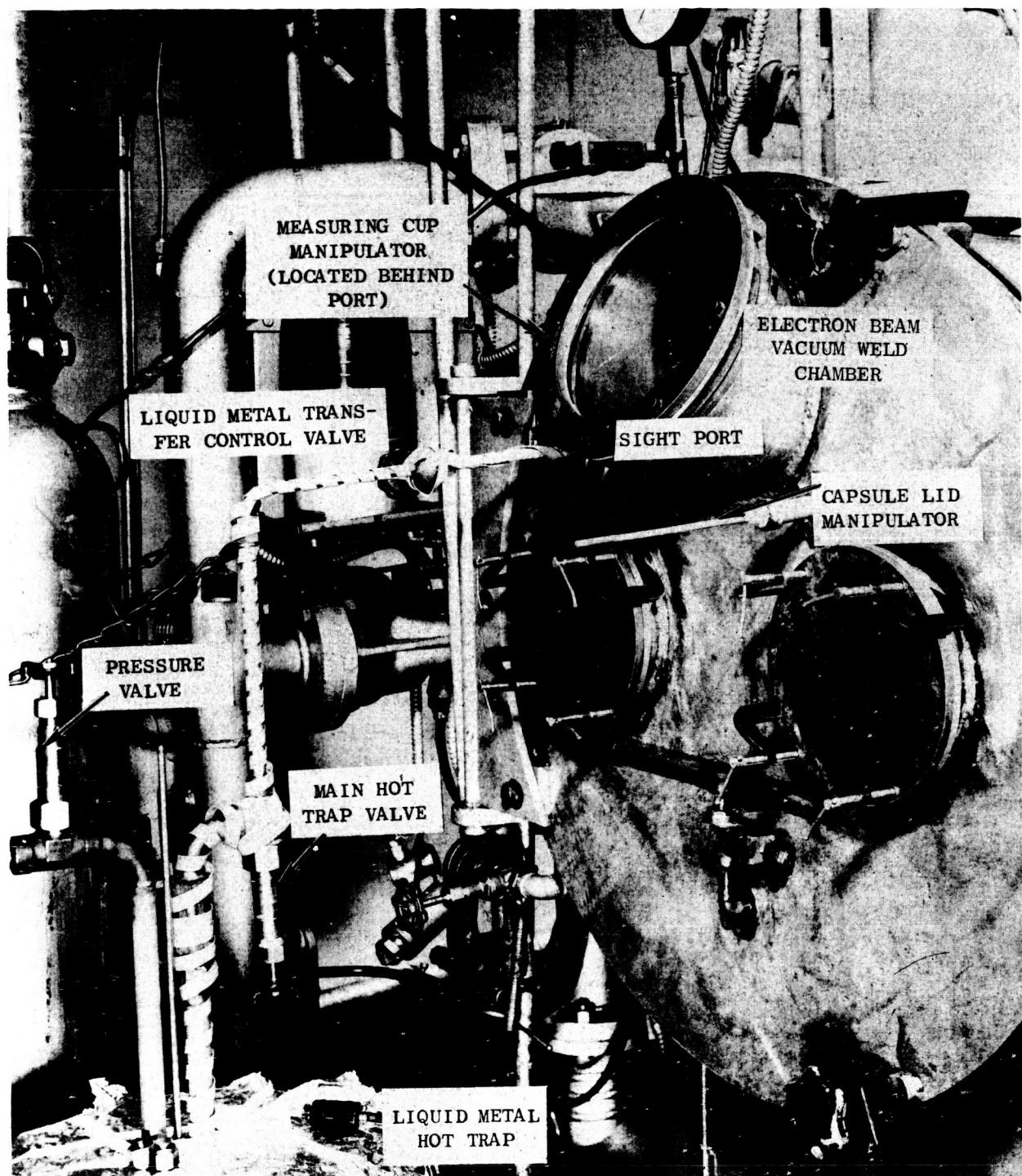


Figure 7. External View of the System for Alkali Metal Transfer. The Hot Trap is Shown Attached to the Vacuum Tank Door. (C63041623)

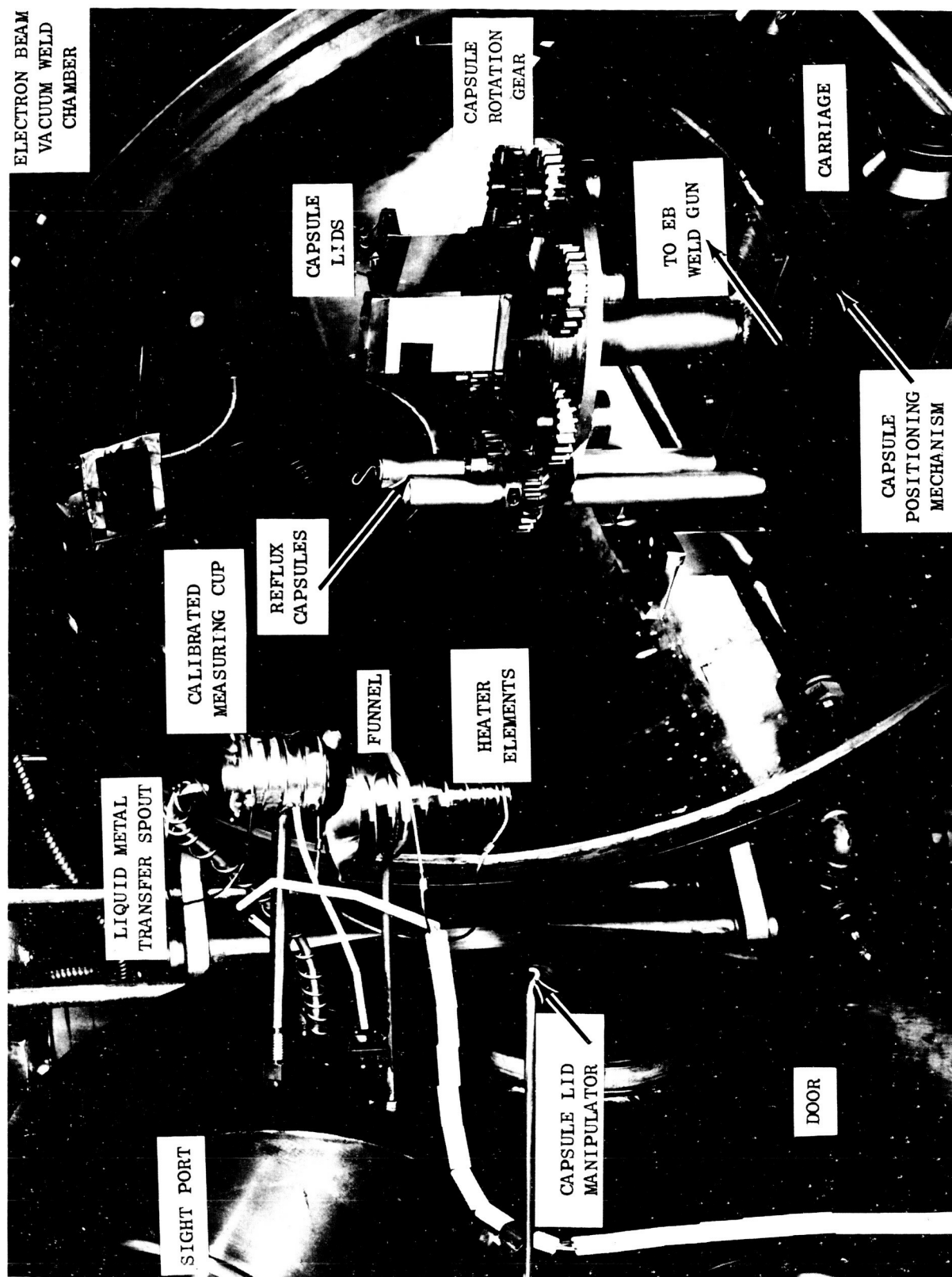


Figure 8. Internal View of System for Alkali Metal Transfer Showing the Necessary Components for Filling of Reflux Capsules. System was Used for Filling Capsules #2, #4, #11 and #12 with Potassium. (C63041622)

using a vacuum rotary feedthrough. After the capsule lid is manually placed on the capsule with the manipulator, the capsule is automatically positioned under the EB gun on the motor driven carriage and the lid is seal-welded to the top of the capsule as the capsule is rotated on its own axis. A series of six capsules can be filled without breaking the vacuum.

The remaining two AS-55 alloy capsules (#11 and #12) and the two D-43 alloy capsules (#2 and #4) were TIG welded, leak checked and cleaned in a similar manner as the first two AS-55 alloy capsules (#8 and #9) and the two Cb-1Zr alloy capsules (#6 and #7) with the exception that fill tubes were not required on these capsules. Bend specimens, 0.060-inch thick x 0.5-inch wide x 2-inch long, also were placed in these capsules. Subsequently, the four capsules were charged with slagged, filtered and hot trapped potassium from shipping container No. 137 using the newly developed vacuum filling system and the procedures that were described in the preceding paragraph.

Analyses of the as-received potassium from container No. 137 used to fill capsules #11, #12, #2 and #4 were supplied by the MSA Research Corporation and are shown in Table IV. Prior to filling the corrosion test capsules, six additional analyses of the as-received potassium were performed for oxygen at General Electric, two by the zirconium-gettering technique, as previously described, and four by the mercury amalgamation method. These data are listed in Table IV and Table II of Appendix B. The method of sampling used for these analyses consisted of filling three separate lengths of 0.5-inch diameter stainless steel pipe, one end of which was attached to the shipping container and the open end was exposed to flowing argon. After allowing approximately 250 grams of potassium to flow through the pipe, the contained potassium was permitted to solidify in two of the pipes and the ends capped in air. Valves had been installed on each end of the third length of pipe and in this case the sample was obtained without exposing the ends to air. The latter pipe and one of the pipes that was capped in air were transferred to the electron beam welding facility and after evacuation and backfilling with argon, the potassium was melted and cast into the Cb-1Zr alloy gettering capsules which were subsequently sealed under vacuum by electron beam welding. The potassium in the remaining length of pipe that was capped in air was analyzed by the mercury amalgamation method. The oxygen content as determined by the zirconium-getter technique again is consistently higher than that determined by the amalgamation method and is attributed to contamination from oxygen in the argon used in the welding chamber during transfer of the potassium to the gettering capsules. Note that there is little difference in the oxygen



results obtained by the zirconium gettering method as a function of the sampling method. However, the oxygen values from both methods are considerably higher than the 17 ppm reported by MSA.

In order to assure that high-purity potassium be used in the remaining capsule corrosion tests, approximately 16 pounds of the as-received potassium from container No. 137 were re-hot trapped at General Electric in a titanium-lined, titanium-gettered container for 200 hours at 1300°F prior to being used to fill the capsules. The oxygen content of the potassium which was sampled from the hot trap after the hot trapping operation was about 21 ppm as analyzed by the mercury amalgamation method.

Additional chemical analyses of metallic impurities also were obtained on the potassium from shipping container No. 137 at an independent laboratory, both before and after the 200-hour, 1300°F hot trapping operation; these values are recorded in Table IV.

In an independent study at this laboratory to evaluate the accuracy of the mercury amalgamation method for oxygen in potassium, three samples were obtained from shipping container No. 137 in lengths of stainless steel pipe which had been evacuated and outgassed before filling with potassium and subsequently capped in air. One sample was analyzed at General Electric by the mercury amalgamation method and two were sent to General Atomics to be analyzed by the neutron activation method where the potassium was melted and transferred to the copper capsules under purified helium<sup>5</sup>. The results are shown in Table IV and are in relatively good agreement. It should be noted that the handling procedures used in obtaining the earlier samples from shipping container No. 137 are suspect and for this reason the latter results are believed to be more representative of the actual oxygen content of the as-received potassium.

To determine whether the re-hot trapped potassium was contaminated during the transfer and filling of the reflux corrosion capsules using the vacuum capsule filling facility, two samples were taken for the analyses of oxygen at the same time that the capsules were filled. One sample tube was filled with potassium on the inside of the chamber in the same manner in which the corrosion capsules were filled and another potassium sample was taken from the transfer line outside of the chamber immediately above the hot trap container. The results obtained on these samples by the mercury amalgamation method are listed in Table IV.

A 1-inch diameter x 10-inch long ancillary Cb-1Zr alloy capsule containing zirconium getter material also was charged with potassium during the actual filling of the corrosion capsules and sealed in an identical manner as the test capsules and the sample tube used for the mercury amalgamation analyses. After a 100-hour exposure at 1400°F, the zirconium getter in the ancillary capsules was analyzed for oxygen, nitrogen and hydrogen by the vacuum fusion techniques. From these data, recorded in Table IV of this section of the report and Table III of Appendix B, the calculated oxygen level in the potassium was found to be 22 ppm which is in good agreement with the results obtained by the mercury amalgamation method, i.e., 33 to 50 ppm. Therefore, it is believed that the oxygen content of the potassium in capsules #11, #12, #2 and #4 was less than 50 ppm.

After filling with about 30 grams of potassium, all eight capsules were enclosed in tantalum foil and pre-tested in a vacuum of about  $4 \times 10^{-5}$  torr at 2000°F for 15 minutes to insure capsule integrity. Subsequently, the eight capsules were grit blasted with -280 mesh alundum powder, using an air pressure of 50 psig, to obtain a high surface emittance.

### C. Testing Procedures

Three tantalum-clad, Pt vs Pt+13%Rh,  $\text{Al}_2\text{O}_3$  insulated thermocouples\* were attached to each capsule, i.e., one at the top, one at the radiating zone and one on the bottom immediately below the heater zone. Thermocouple placement, heater location and radiation shielding are shown on drawings of the capsules in Figures 9 and 10 and in the photograph of the capsule assembly in Figure 11.

Four instrumented reflux capsules (#6, #7, #8 and #9) and their respective heaters and shielding were secured in a Varian ultra-high vacuum chamber, C-III. This type of vacuum equipment consist of an 18-inch diameter x 30-inch high bakeable chamber connected to a 400  $\text{Q/sec}$  getter-ion pump and three cryogenic molecular sieve roughing pumps, Figure 12. Initial evacuation of the chamber to about two torr is accomplished with a mechanical pump and subsequently to the  $10^{-3}$  torr range with the cryogenic adsorption pumps. Final evacuation to the ultimate pressure in the  $10^{-10}$  torr range is accomplished

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\* Purchased from Thermo Electric Company, Inc.; typical purity of the  $\text{Al}_2\text{O}_3$  is 99.65% with the following maximum impurity levels:  $\text{SiO}_2$ , 0.08%;  $\text{Fe}_2\text{O}_3$ , 0.10%;  $\text{Na}_2\text{O}$ , 0.06%;  $\text{MgO}$ , 0.08%;  $\text{CaO}$ , 0.08%;  $\text{ZrO}_2$ , 0.08%; C, 0.01%; B, 0.0001%.

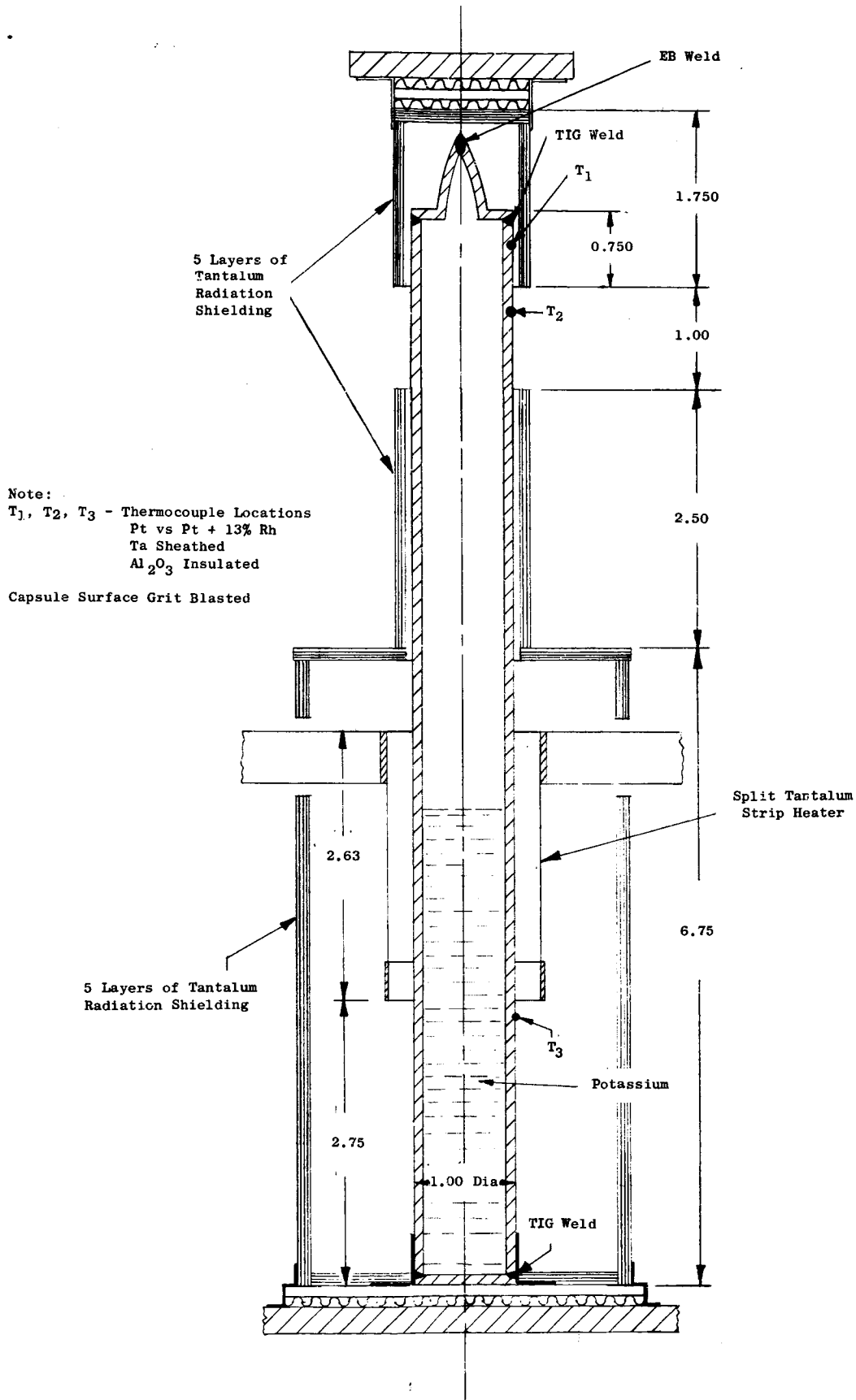


Figure 9. Drawing of Reflux Capsule Illustrating Location of Radiation Shielding, Radiation Zone, Tantalum Heating Elements, and Height of Potassium at Temperature in Capsules #6, #7, #8 and #9.

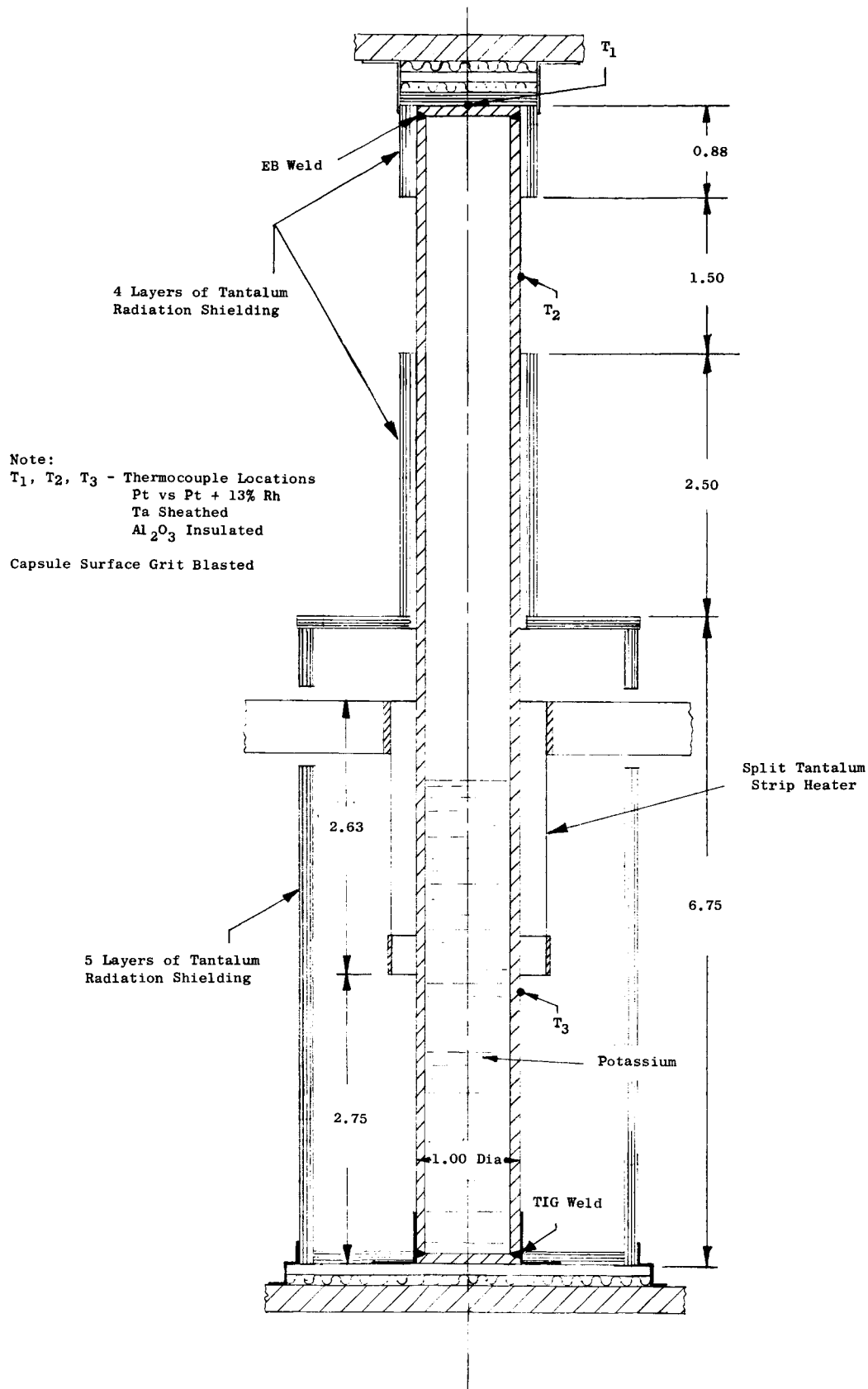


Figure 10. Drawing of Reflux Capsule Illustrating Location of Radiation Shielding, Radiation Zone, Tantalum Heating Element, and Height of Potassium at Temperature in Capsules #2, #4, #11 and #12.

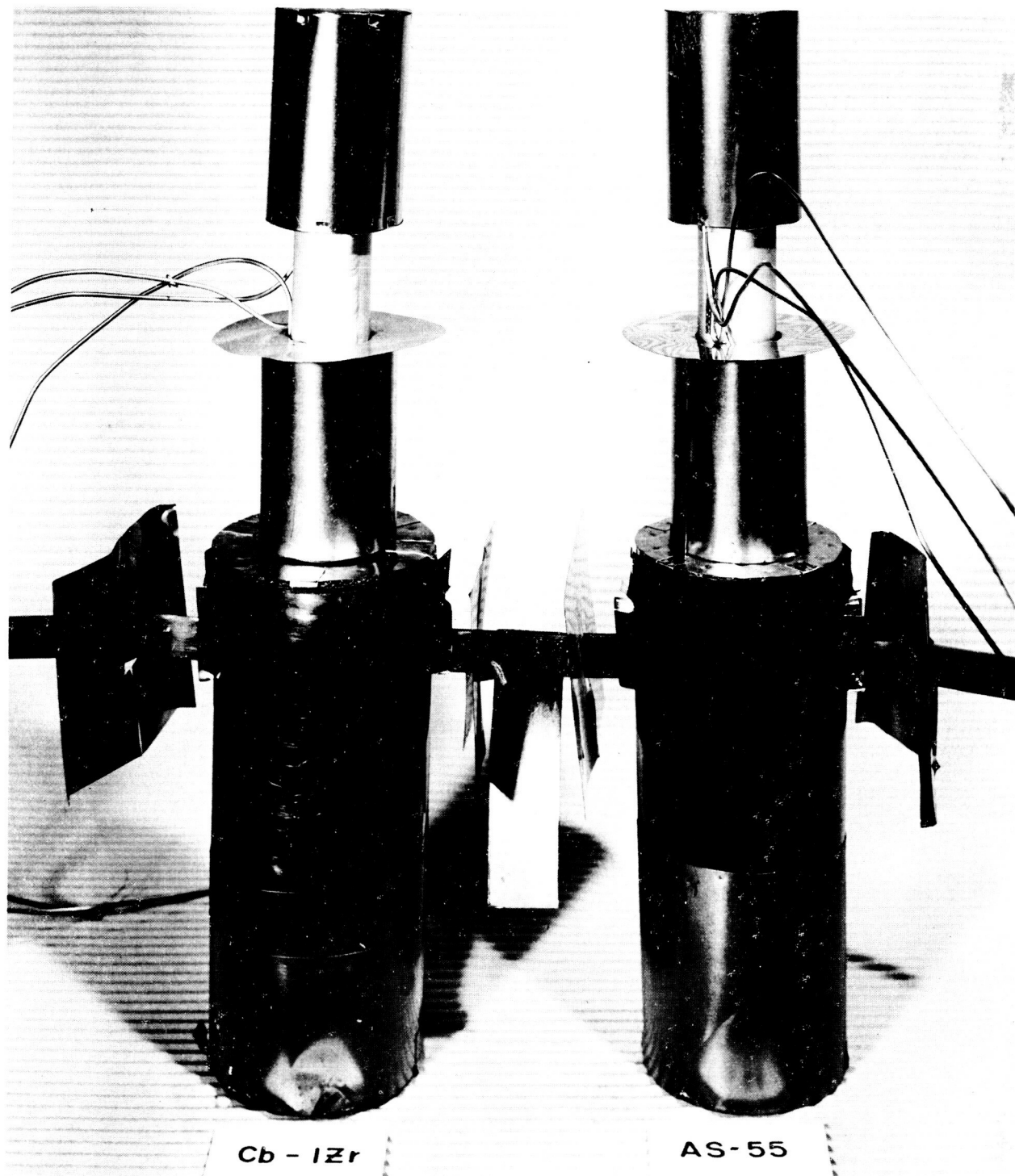


Figure 11. AS-55 and Cb-1Zr Alloy Reflux Corrosion Capsules Containing Potassium and with Tantalum Radiation Heaters and Shielding in Place. (C63052103)

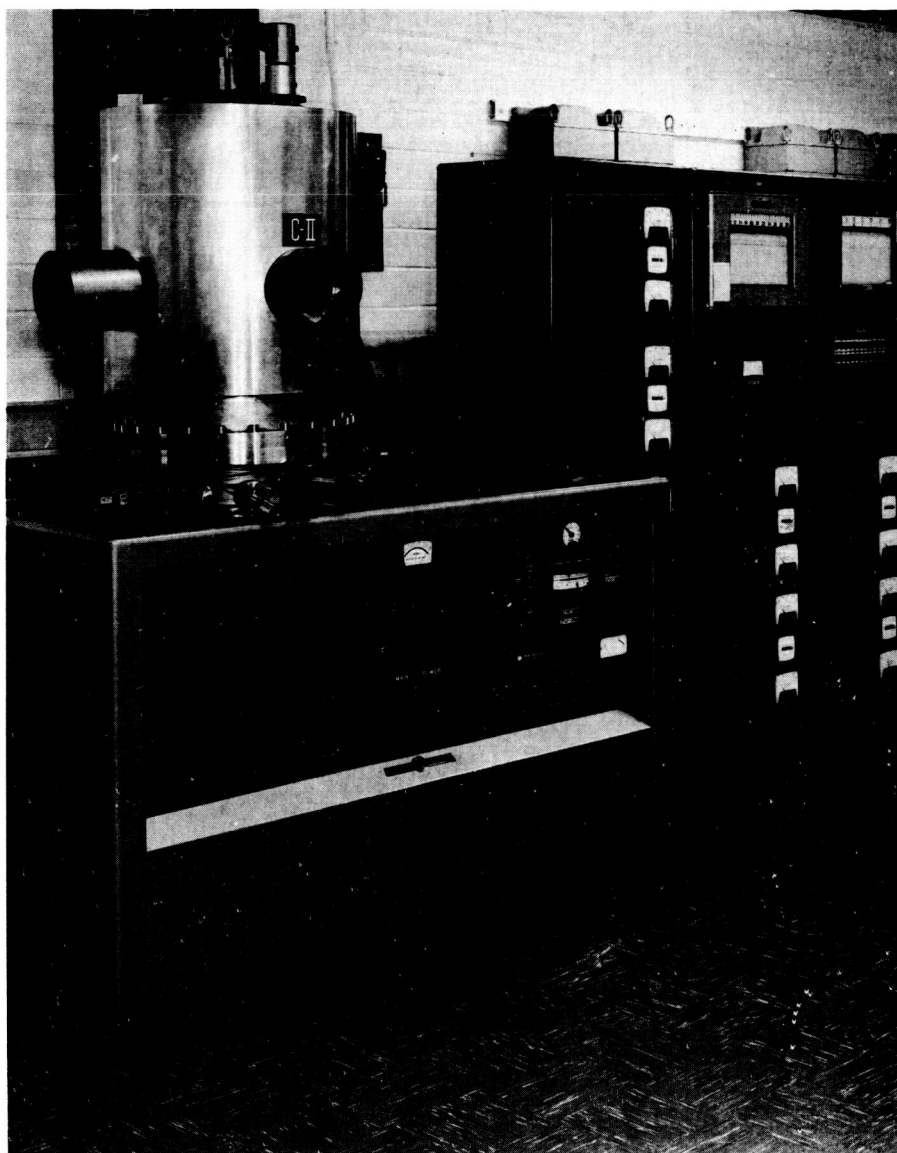


Figure 12. Ultra High Vacuum System' ( $10^{-10}$  Torr Range) Used in the Study of the Corrosion Behavior of AS-55, D-43, and Cb-1Zr Alloys in Potassium. The Chamber is 18 Inches in Diameter and 30 Inches High and Incorporates a 400  $\text{l}/\text{Sec}$  Getter-Ion Pump and 3 Adsorption Pumps. (C64051216)

with the getter-ion pump after a suitable bakeout period. Pressure is measured by a Bayard-Alpert type ionization gauge attached to the side of the chamber.

After installing capsules #6 and #7 (Cb-1Zr alloy) and #8 and #9 (AS-55 alloy) in the vacuum chamber, the chamber was evacuated to a pressure of  $7.0 \times 10^{-7}$  torr and baked out at  $400^\circ\text{F}$  for 8 hours after which the pressure reached  $7.0 \times 10^{-7}$  torr. Subsequently, the capsules were heated to  $2000^\circ\text{F}$  with the pressure being held below  $3.0 \times 10^{-6}$  torr during the entire heat-up cycle. Although some unstable boiling was evident during heat-up, the instability appeared to subside when the test temperature was reached. The four capsules were held at the test temperature of  $2000^\circ\text{F}$  for 5,000 hours. The pressure profile over the 5,000-hour test period is shown in Figure 13. One pressure rise was encountered at the end of approximately 4,500 hours when the coolant water to the vacuum bell jar was accidentally turned off. The pressure reached a maximum of  $7.0 \times 10^{-7}$  torr for a short period of time and dropped quickly when the coolant water was restored. At the end of the 5,000-hour exposure, capsules #6 (Cb-1Zr alloy) and #8 (AS-55 alloy) were removed from the chamber and capsules #7 (Cb-1Zr alloy) and #9 (AS-55 alloy) were put back on test at  $2000^\circ\text{F}$  to complete the 10,000 hours of testing.

Temperature control was accomplished by using a voltage regulated, variac controlled supply to a step-down transformer which fed the power to the tantalum heaters. Two heaters were connected in series so that only two power supplies were required for the testing of the four capsules. The average measured temperatures during testing are shown in Table V. Individual temperature measurements taken during the 5,000-hour exposure of capsule #8 (AS-55 alloy) are plotted in Figure 14. The data show that very little emf drift occurred in the thermocouples over the 5,000-hour exposure and indicate that the tantalum-sheathed,  $\text{Al}_2\text{O}_3$ -insulated Pt vs Pt + 13% Rh thermocouples are quite stable over the temperature range of  $1950^\circ$  to  $2000^\circ\text{F}$ . This is a contrast to data obtained by Oak Ridge National Laboratory<sup>(6)</sup> for tantalum-sheathed, MgO-insulated Pt vs Pt + 10% Rh thermocouples at  $1200^\circ\text{C}$  ( $2192^\circ\text{F}$ ) in a vacuum of  $10^{-7}$  torr. In the latter case a drift of  $-54^\circ\text{C}$  ( $-129^\circ\text{F}$ ) was reported to have occurred in 143 hours.

Reflux capsules #11 and #12 (AS-55 alloy) and #2 and #4 (D-43 alloy) were installed in the Varian ultra-high vacuum chamber C-11, which is similar to chamber C-III, and instrumented in the same manner as were capsules #6, #7, #8, and #9. The chamber was evacuated to a pressure of  $5.0 \times 10^{-7}$  torr and baked out for 12 hours at  $400^\circ\text{F}$ .

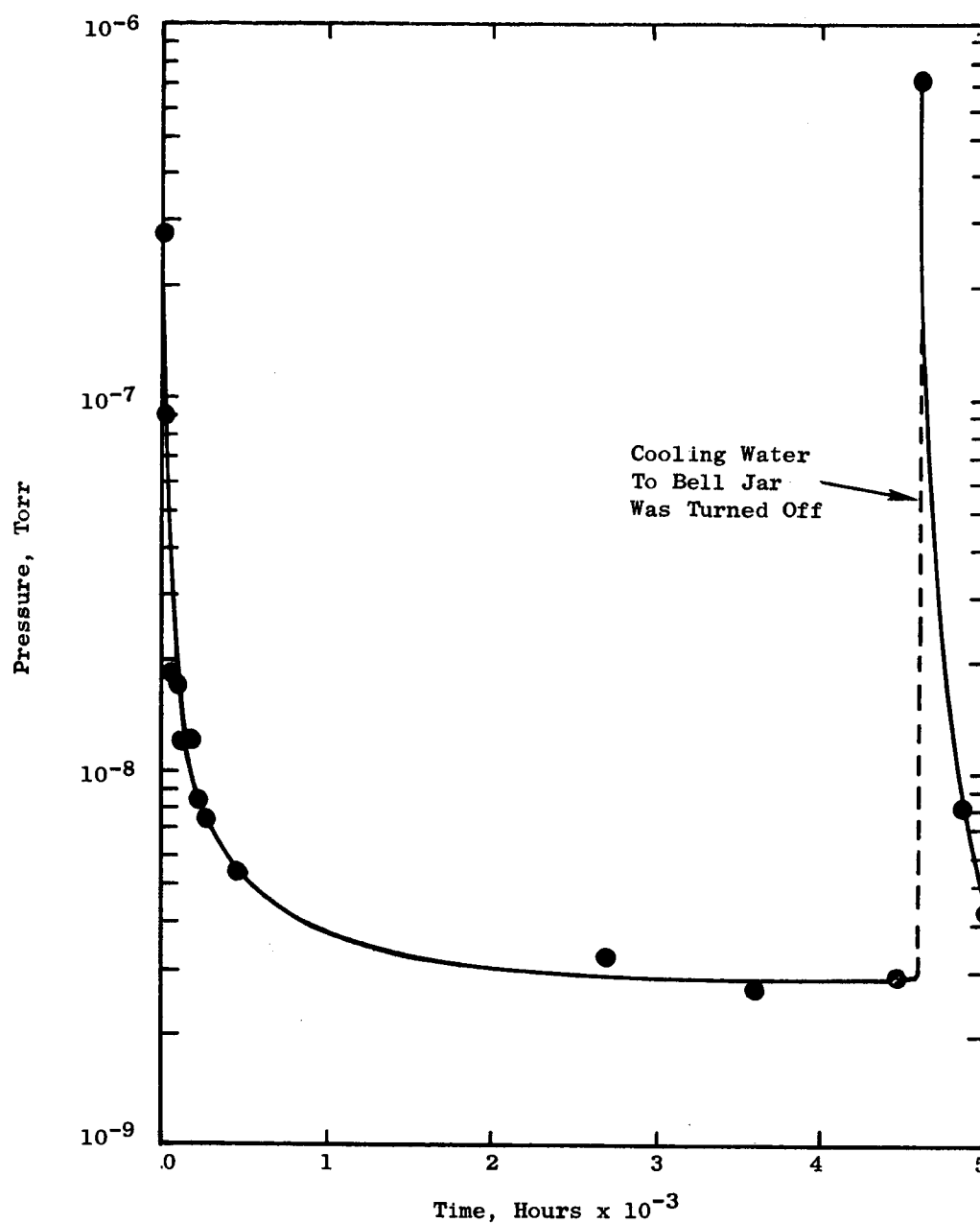


Figure 13. Pressure Profile of High-Vacuum Chamber C-III During Testing of Cb-1Zr Alloy Reflux Corrosion Capsule #6 and AS-55 Alloy Reflux Corrosion Capsule #8 at 2000°F for 5,000 Hours.



TABLE V

## AVERAGE TEST TEMPERATURES OF REFLUX CAPSULES #6 (Cb-12r ALLOY) AND

## #8 (AS-55 ALLOY) DURING 5,000-HOUR EXPOSURE TO POTASSIUM

Capsule No.	Location of Temp. Measurement	Method of Temp. Measurement	Average Temp., °F	Mean Deviation, °F
6	Heater Region	Thermocouple	1919	± 22
	Radiating Region	Brightness Pyro- meter(1)	1862	± 17
	Top Region	Thermocouple	1867	± 15
8	Heater Region	Thermocouple	1985	± 7
	Radiating Region	Thermocouple	1913	± 25
		Brightness Pyro- meter(1)	1890	± 12
	Top Region	Thermocouple	1962	± 10

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(1) Uncorrected for Pyrex Window and Emissivity.

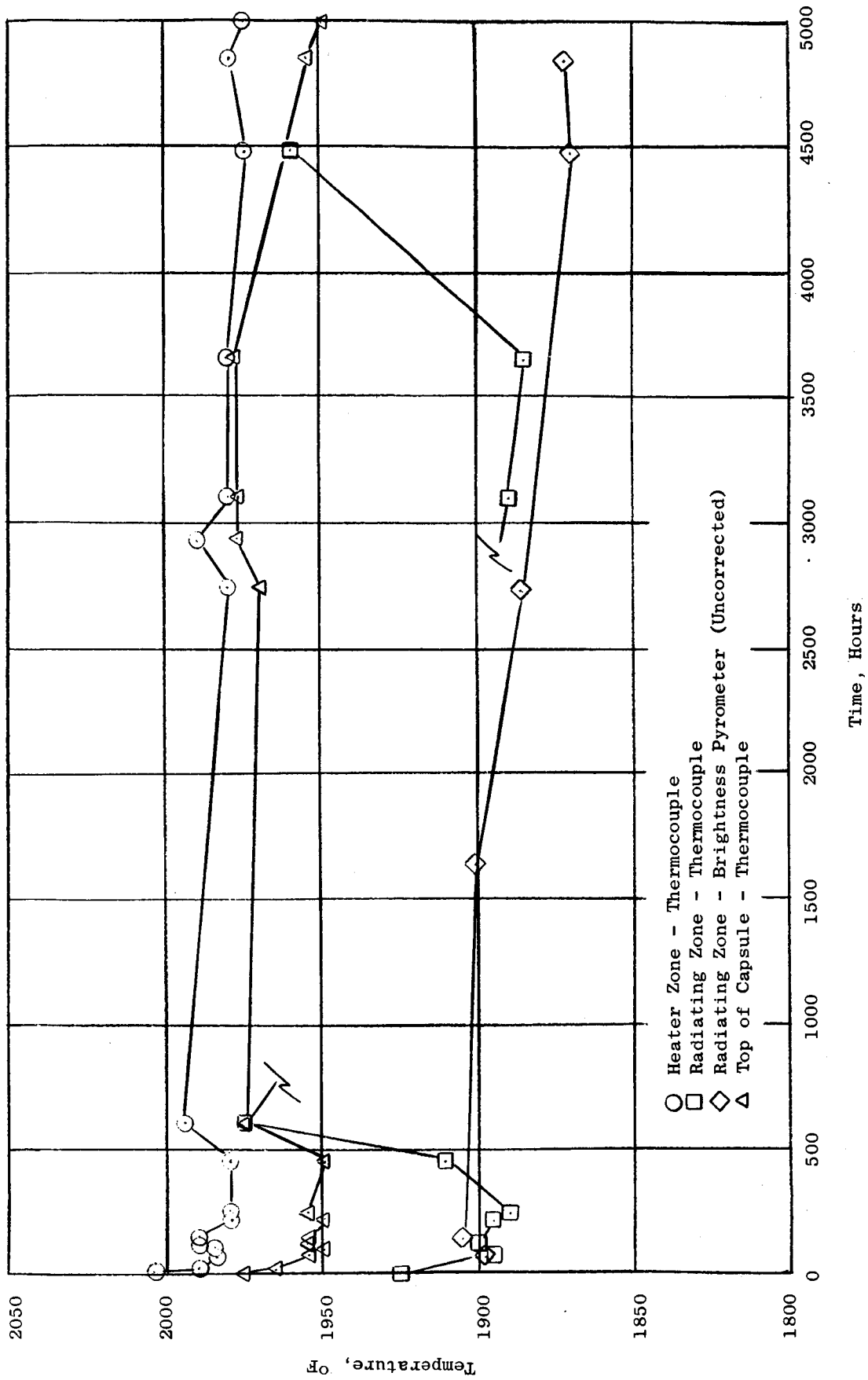


Figure 14. Indicated Temperatures of Reflux Corrosion Capsule #8 (AS-55 Alloy) During 5,000-Hour Exposure to Potassium.

after which the pressure was  $5.4 \times 10^{-9}$  torr, as measured by a Bayard-Alpert type ionization gauge attached to the side of the chamber. During heat-up to the test temperature of 2000°F, the pressure reached a maximum of  $4.4 \times 10^{-6}$  torr. The pressure profile obtained during the test is shown in Figure 15. Note that the chamber was opened after approximately 3,000 hours in order to repair a heater which was shorting to the tantalum shielding. No further difficulty was encountered for the remainder of the test period. At the end of the 5,000-hour exposure, capsules #12 (AS-55 alloy) and #2 (D-43 alloy) were removed from the chamber and capsules #11 (AS-55 alloy) and #4 (D-43 alloy) were put back on test at 2000°F to complete the 10,000 hours of testing.

Temperature control also was accomplished by the same technique as that used for capsules #6, #7, #8 and #9. The average measured temperatures of the capsules #12 and #2 during testing are shown in Table VI.

Two commonly used techniques for determining the reflux rate of potassium in capsules are: 1) a direct calculation of the heat radiated from the condensing region of the capsule to the vacuum chamber wall, and 2) an indirect calculation of the heat radiated from the condensing region by the subtraction of the heat losses occurring in the capsule, in regions other than the condensing region, from the total heat input to the capsule. Because of the difficulties in determining the heat losses of the capsules in the experimental setup described in this report, the heat radiated from the condensing region of the capsule to the vacuum chamber wall was calculated using the first method listed above. The heat flux was calculated using the equation:

$$q_{12} = A_1 \sigma \left[ \frac{1}{\frac{1}{\epsilon_1} + \frac{A_1}{A_2} \left( \frac{1}{\epsilon_2} - 1 \right)} \right] \left[ T_1^4 - T_2^4 \right] \quad (1)$$

Assuming:

1. Coaxial cylinders
2.  $T_2 = 0^\circ \text{R}$

and letting,

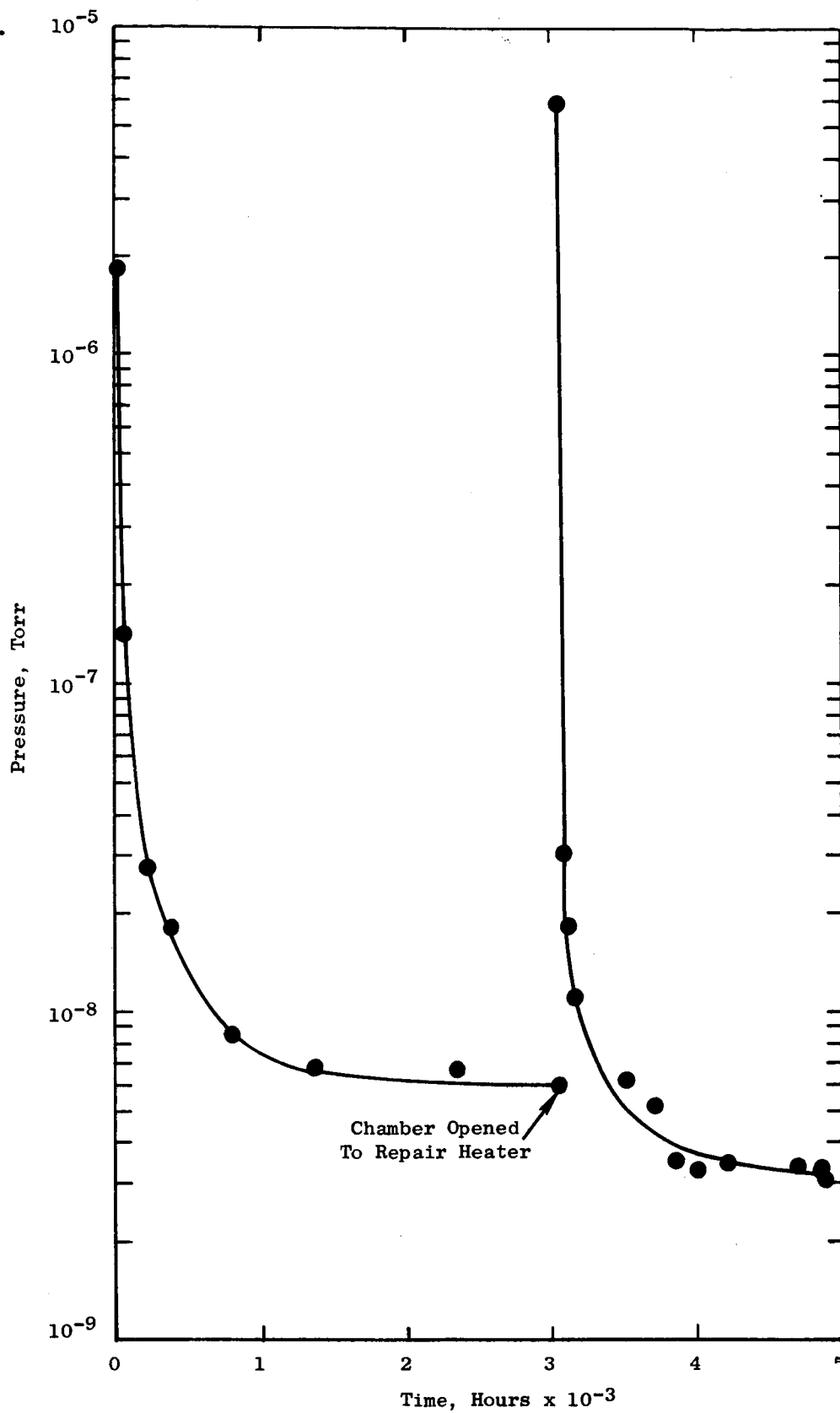


Figure 15. Pressure Profile of High-Vacuum Chamber C-II During Testing of AS-55 Alloy Reflux Corrosion Capsule #12 and D-43 Alloy Reflux Corrosion Capsule #2 at 2000°F for 5,000 Hours.

TABLE VI

## AVERAGE TEST TEMPERATURES OF REFLUX CAPSULES #12 (AS-55 ALLOY) AND

## #2 (D-43 ALLOY) DURING 5,000-HOUR EXPOSURE TO POTASSIUM

Capsule No.	Location of Temp. Measurement	Method of Temp. Measurement	Average Temp., °F	Mean Deviation, °F
12	Heater Region	Thermocouple	1916	$\pm 15$
	Radiating Region	Thermocouple	1901	$\pm 18$
		Brightness Pyrometer <sup>(1)</sup>	1948	$\pm 8$
	Top Region	Thermocouple	-- (2)	-- (2)
2	Heater Region	Thermocouple	1941	$\pm 11$
	Radiating Region	Brightness Pyrometer <sup>(1)</sup>	1962	$\pm 30$
	Top Region	Thermocouple	1805	$\pm 55$
		-----		

(1) Uncorrected for Pyrex Window and Emissivity.

(2) No Reading, Thermocouple Failed.

$$A_1 = 0.0327 \text{ ft}^2 \text{ (radiating area of capsules)}$$

$$A_2 = 7.85 \text{ ft}^2 \text{ (absorbing area of chamber)}$$

$$\epsilon_1 = 0.4 \text{ (emissivity of capsule)}$$

$$\epsilon_2 = 0.2 \text{ (emissivity of chamber)}$$

$$T_1 = 2410^\circ \text{R (temperature of capsule surface)}$$

$$\sigma = 0.174 (10^{-8}) \text{ BTU hr}^{-1} \text{ ft}^{-2} \text{ }^\circ\text{F}^{-4} \text{ (Stefan-Boltzmann's constant)}$$

then,

$$q_{12} = 762 \text{ BTU/hr}$$

The refluxing rate  $W$  (lbs/hr/ft<sup>2</sup>) is calculated from the equation:

$$W = \frac{q_{12}}{H_v A_3} \quad (2)$$

where,

$$H_v = 745 \text{ BTU/lb for potassium at } 1950^\circ\text{F}^7$$

$$A_3 = 0.0275 \text{ ft}^2 \text{ (area of condensing surface).}$$

Then,

$$W = 37.2 \text{ lbs/hr/ft}^2$$

However, the reflux rate can vary significantly with variations in the measured and assumed test conditions. By letting the emittance of the radiating surface,  $\epsilon_1$ , vary from 0.3 to 0.5, the emissivity of the chamber wall,  $\epsilon_2$ , vary from 0.1 to 0.3 and the temperature of the radiating surface vary from 1900° to 2000°F, it is possible for the reflux rate to vary from a minimum of 25.2 lbs/hr/ft<sup>2</sup> to a maximum 51.1 lbs/hr/ft<sup>2</sup>. Further, if it is assumed that half of the radiating surface of the capsule is exposed to a heat sink that is at a temperature of approximately 1000°F, which is probably more realistic for the case in the test setup for these capsules, similar calculations using equations (1) and (2) show the possible range for the reflux rate to be 23.4 lbs/hr/ft<sup>2</sup> to 48.0 lbs/hr/ft<sup>2</sup>. Overall, it is believed that the refluxing rate was about  $37 \pm 12$  lbs/hr/ft<sup>2</sup>.

It should be noted that in more recent work at General Electric under NASA sponsorship<sup>8</sup>, a facility has been built and operated which provides a more accurate determination of the heat flux from the condensing region of the capsule than either of the previously discussed techniques. This facility utilizes a heat exchanger around the condensing region of the capsule. The heat from the capsule is radiated to the heat exchanger and is transferred to a flowing water system, thus allowing an accurate heat balance to be performed on the water system.

#### D. Results of 5,000-Hour Reflux Capsule Corrosion Tests

With the exception of the AS-55 alloy capsule #12, no changes were noted in the external appearance of the capsules as a result of the 5,000-hour exposures in vacuum at 2000°F. In the case of the AS-55 alloy capsule #12, a portion of the tantalum thermocouple sheath was observed to have bonded to the side of the capsule wall, Figure 16. Upon sectioning the capsules, white deposits were found on the inside surface of the AS-55 alloy capsule #12 on the opposite side of the wall where the tantalum thermocouple sheath had bonded, Figure 17, and also on the surface of the weld at the bottom of the capsule. Because of the extremely small quantity of the deposit, an x-ray diffraction analysis was unsuccessful. However, a spectrographic analysis showed the deposit to contain yttrium. The deposit presently is believed to be yttrium oxide.

An examination of the microstructure of a cross section of the capsule wall through the area where the thermocouple sheath had bonded is shown in Figure 18; no variation in the microstructure of the AS-55 alloy was observed in the area of the tantalum bond. However, the hardness of the AS-55 alloy in the area of the tantalum bond increased from 140 KHN to 175 KHN. This increase in hardness was observed to a depth of 6 mils and is attributed to diffusion of oxygen from the impurities in the Al<sub>2</sub>O<sub>3</sub> thermocouple insulation into the AS-55 alloy. The defect observed on the inner surface of the capsule wall, as shown in Figure 18, is believed to have been caused in the original capsule forming operation as evidenced by the localized cold work around the deformed area. No other significant changes in appearance were observed on the inside of the AS-55 alloy capsule #12.

Visual examination of the inside surfaces of the AS-55 alloy capsule #8 and Cb-1Zr alloy capsule #6, Figures 19 and 20, revealed:

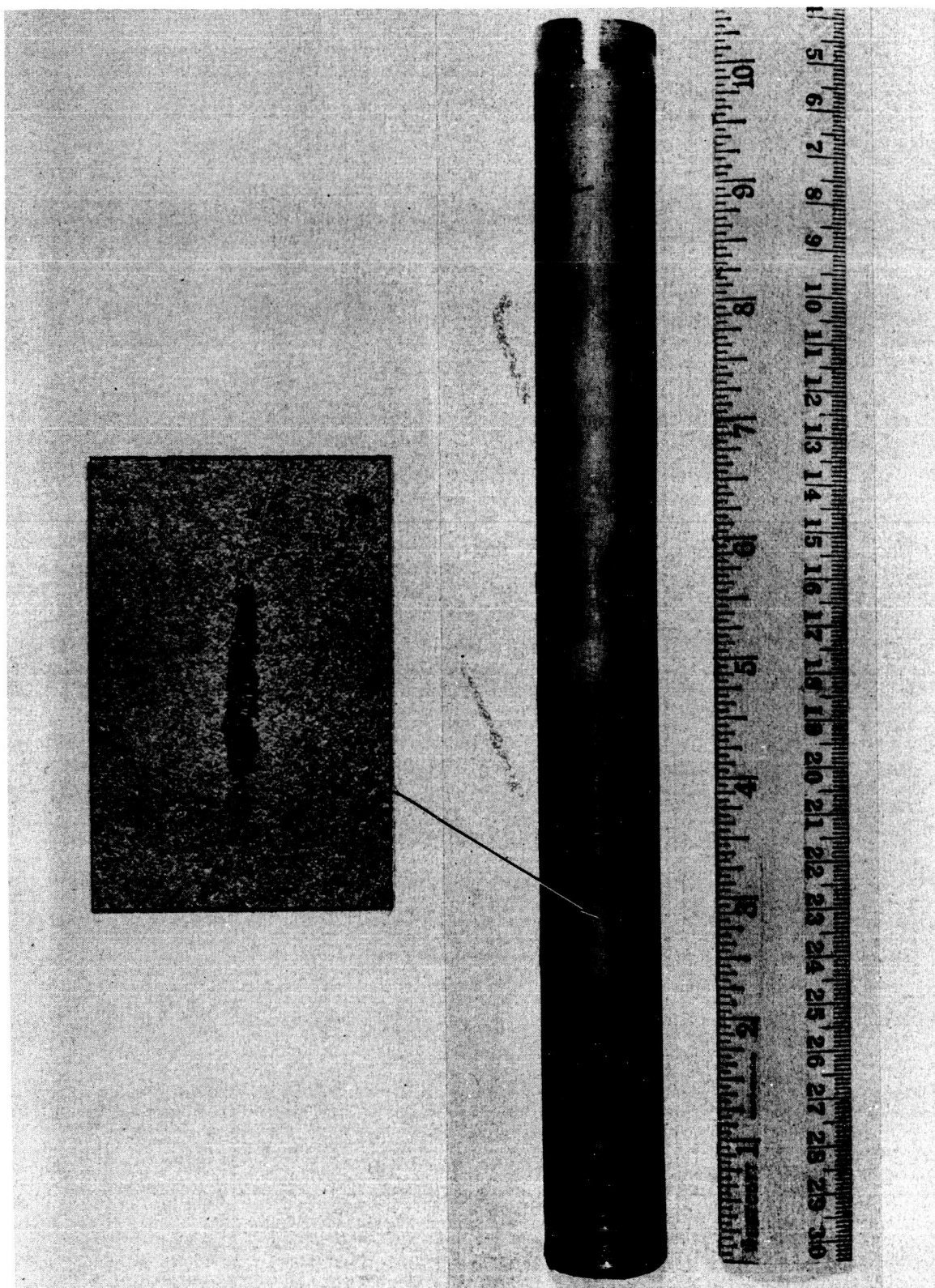


Figure 16. AS-55 Alloy Reflux Corrosion Capsule #12 After a 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr. Note Bonding of Tantalum Thermocouple Sheath to Capsule at Thermocouple Junction. Left: Mag: 16X (C64012206); Right: (C64012208)



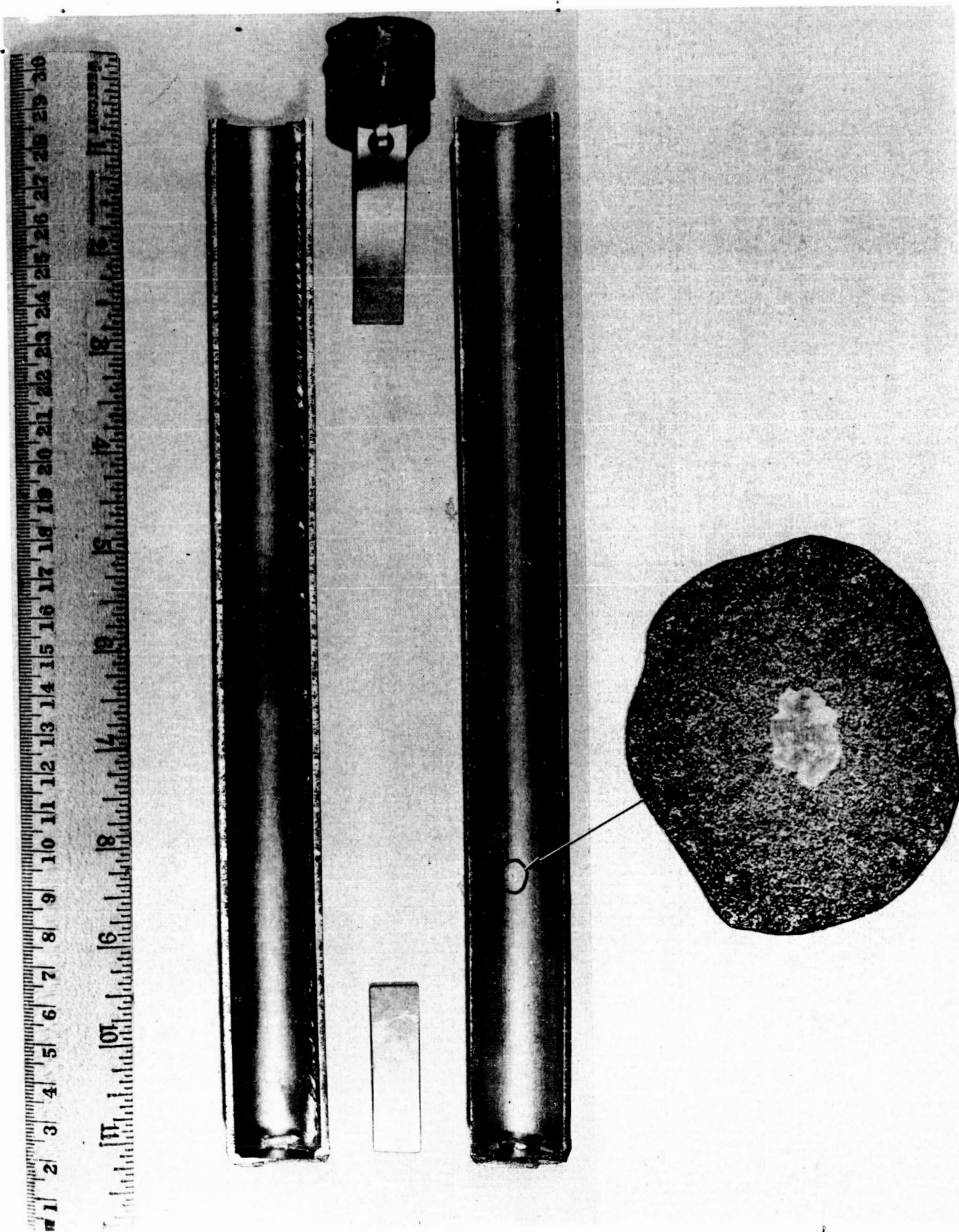
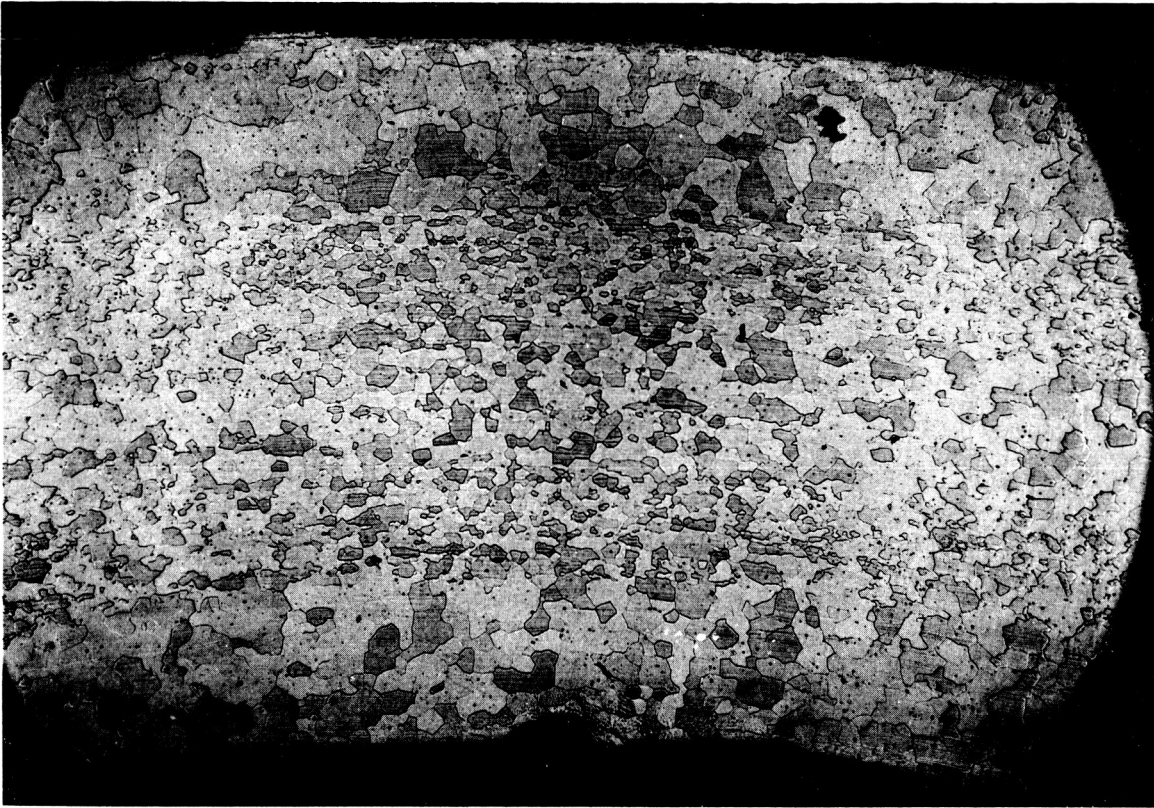


Figure 17. Sectioned AS-55 Alloy Reflux Corrosion Capsule #12 After a 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr. Note the Deposit on the Inside Wall Opposite the Location of the Thermocouple Junction Bond. Right: Mag: 16X (C64012205); Left: (C64012207)

External  
Surface



Internal  
Surface

Figure 18. Microstructure of Transverse Section Through Liquid Region of AS-55 Reflux Corrosion Capsule #12 After a 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr. Note Bonding of Tantalum Thermocouple Sheath on External Surface. Mag: 50X (K4708) Etchant: 20% $\text{Hf}$ -20% $\text{HNO}_3$ -60% $\text{H}_2\text{O}$ .

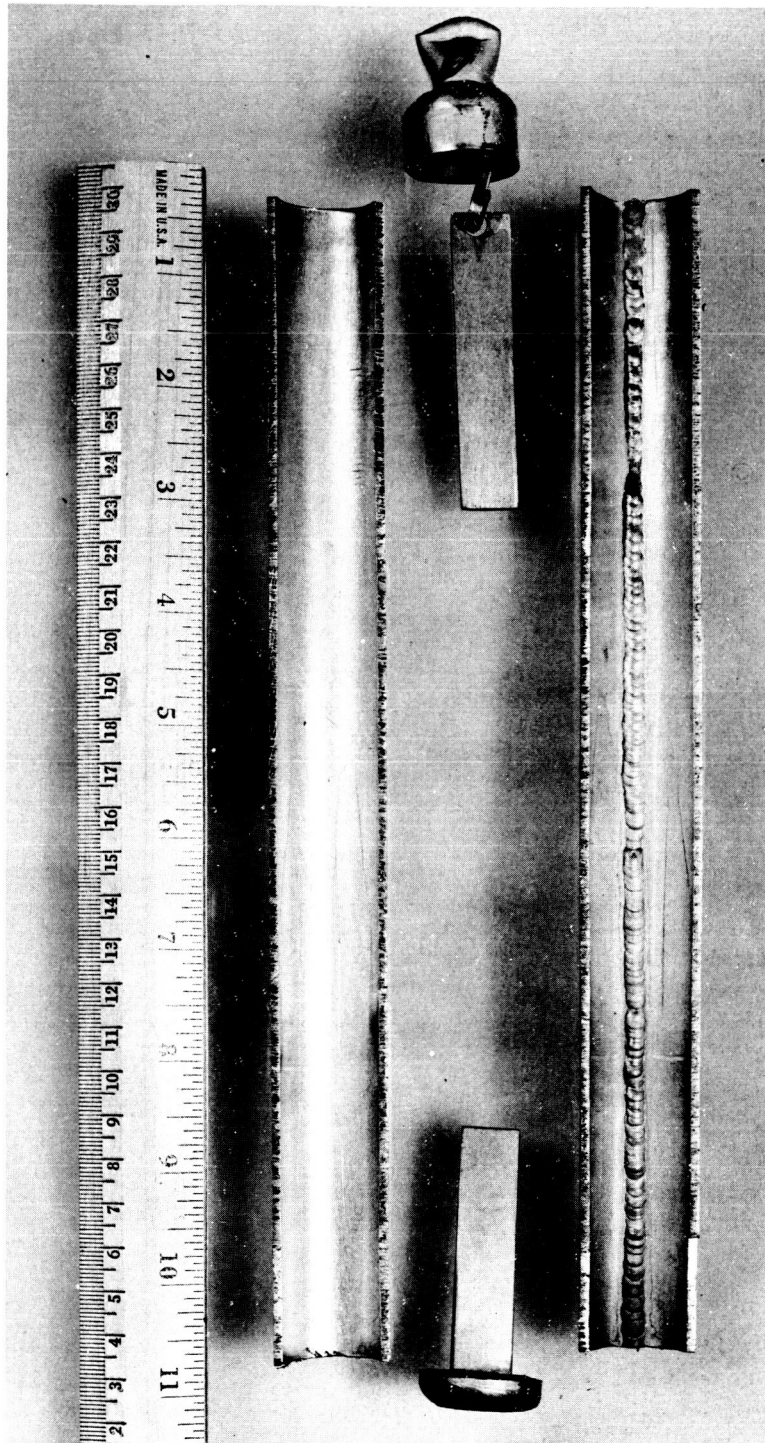


Figure 19. Sectioned AS-55 Alloy Reflux Corrosion Capsule #8 After a 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr. (C63071113)

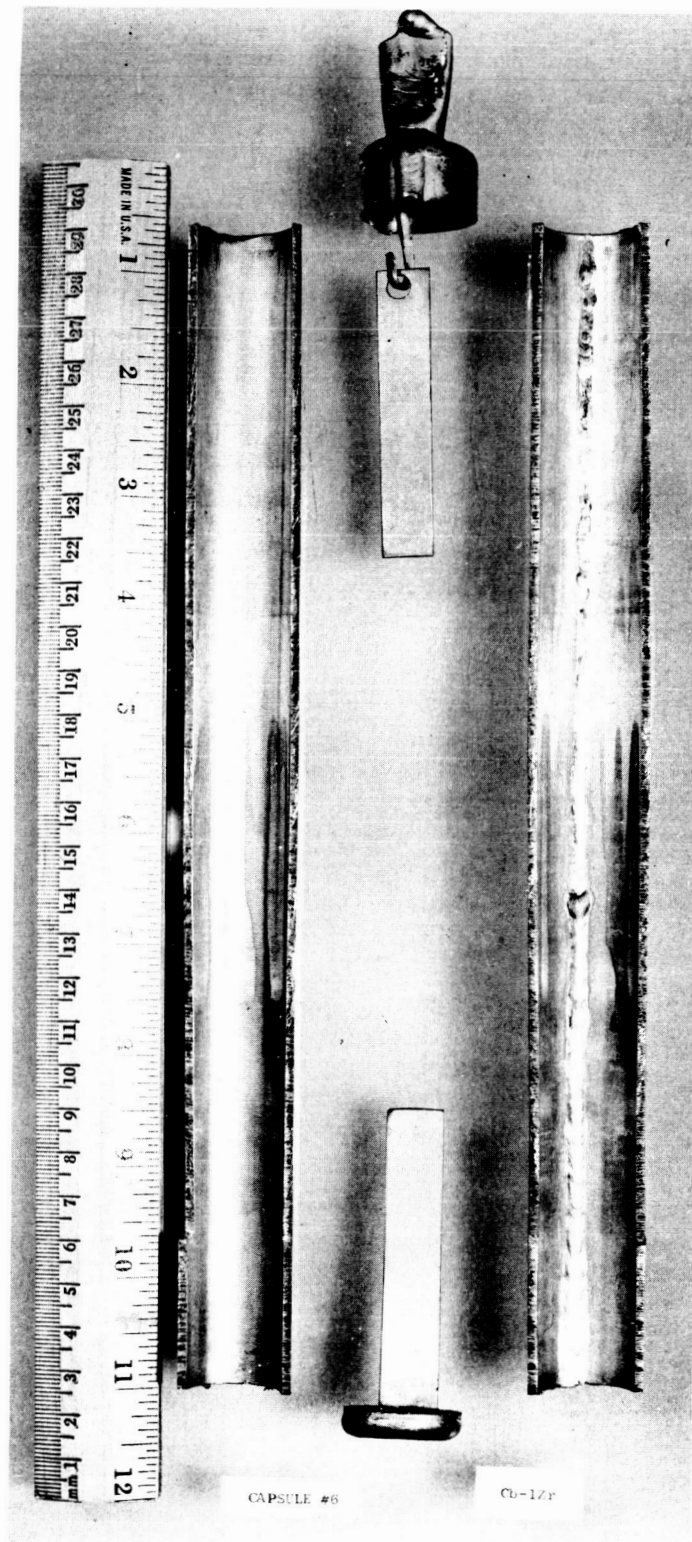


Figure 20. Sectioned Cb-1Zr Alloy Reflux Corrosion Capsule #6 After a 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr. (C63071114)

1. Discolorations in the region between the primary condensing zone and the liquid zone,
2. a change in surface appearance in the heat affected-zone of the weld, and
3. in general, a more lustrous appearance above the liquid region.

It should be noted that a major difference exists between the two AS-55 alloy capsules #12 and #8 in that two different potassium filling techniques were used in the preparation of the capsules. Capsule #12 was filled using the vacuum transfer facility and the data indicated that the oxygen content in the potassium in capsule #12 was considerably lower than the oxygen content in the potassium in capsule #8. Capsule #6 (Cb-1Zr alloy) was filled using the same facility and potassium as capsule #8 (AS-55 alloy). The holes that are observed in the weld beads of capsule #8, Figure 19, were in the weld prior to testing as confirmed by pre-test radiographic inspection. Their presence is attributed to improper weld termination and subsequent shrinkage during solidification of the weld.

The D-43 alloy capsule #2 showed no significant changes in appearance of the internal surface other than having a uniform lustrous surface, Figure 21. This capsule was filled with potassium using the same technique as used in filling AS-55 capsule #12, i.e., the vacuum transfer facility.

In addition to visual examination, the following tests were conducted on specimens machined from the capsule wall, Figure 22, in an attempt to determine the effect of the 5,000-hour exposure to the potassium and the ultra-high vacuum on the capsule material:

1. Chemical analyses
2. Metallographic examinations
3. Hardness measurements
4. Weight and thickness measurements
5. Bend tests on samples contained with the capsule
6. Stress-rupture tests at 2000°F on specimens machined from the AS-55 alloy and D-43 alloy capsule walls.



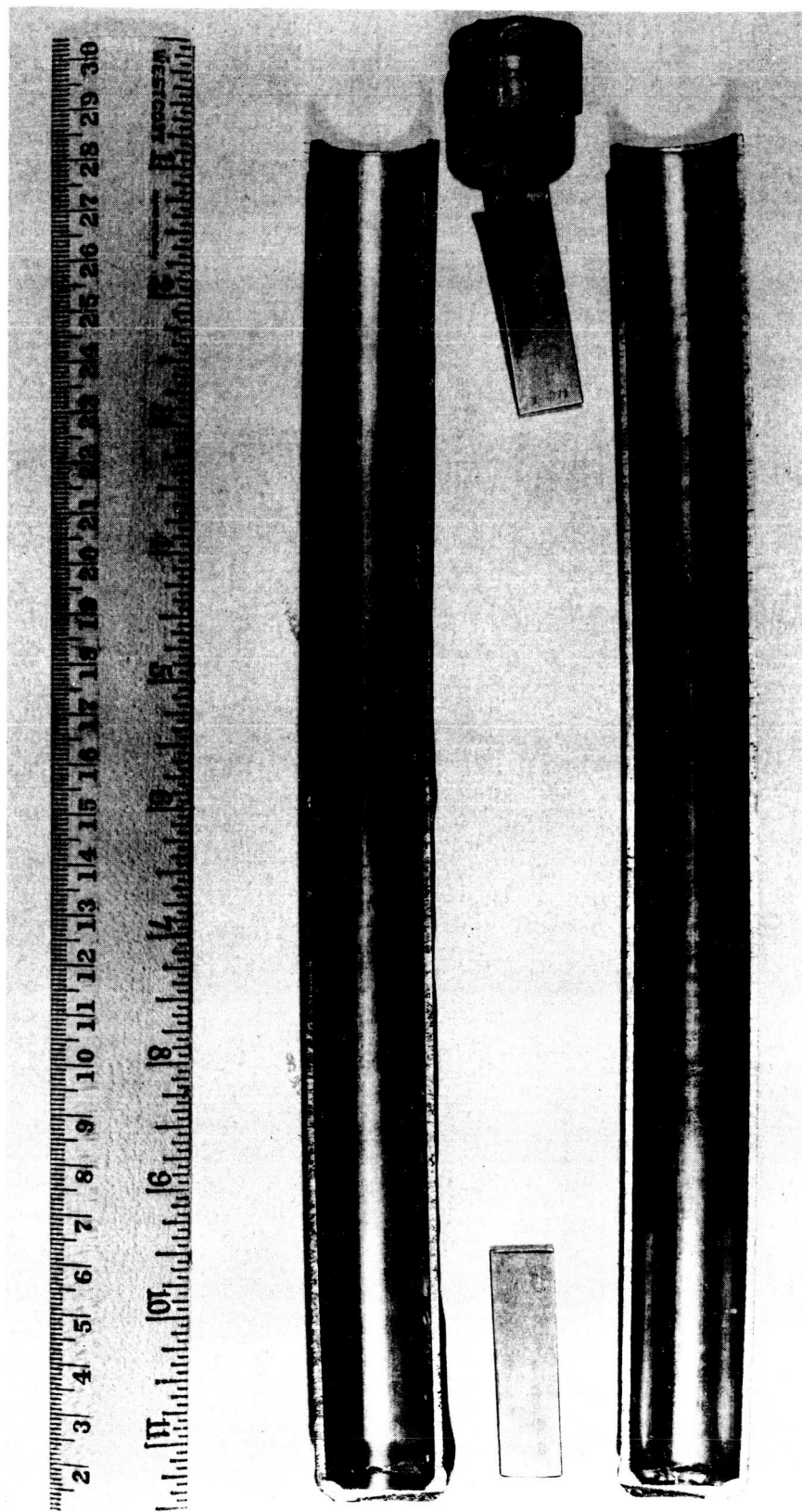
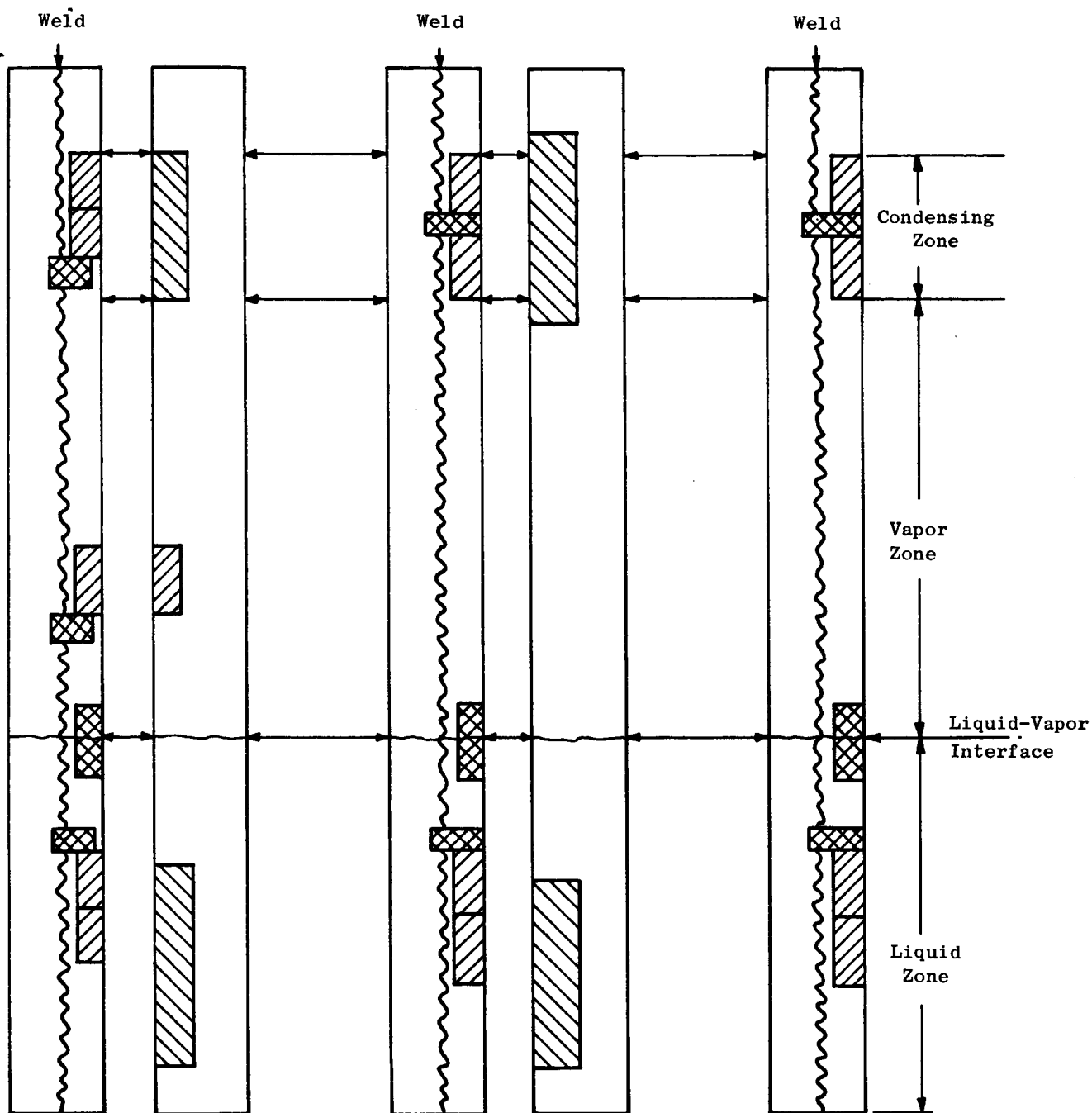


Figure 21. Sectioned D-43 Alloy Reflux Corrosion Capsule #2 After a 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr. (C64012209)



Capsule #6 Cb-1Zr  
Capsule #8 AS-55

Capsule #2 D-43

Capsule #12 AS-55

▨ Chemistry Samples    ▩ Metallographic Samples    ▧ Stress-Rupture Samples

Figure 22. Location of Samples from Wall of Reflux Corrosion Capsules for Chemical Analyses, Metallographic Examination and Stress-Rupture Tests.

## 1. Chemical Analyses

Results of chemical analyses of samples taken at locations shown in Figure 22 are recorded in Table VII. To determine the chemical composition of the internal and external surfaces, two 0.080-inch thick specimens were taken from the wall of the capsules at each location. The inside 0.040 inch of material was removed from one specimen, the outer 0.040 inch of material was removed from the second specimen, and the remaining material analyzed for oxygen, nitrogen, hydrogen and carbon.

No significance is attached to changes observed in the nitrogen, carbon or hydrogen contents of any of the capsules. Good agreement was found in the pre- and post-test nitrogen analyses for capsules #6 (Cb-1Zr alloy) and #2 (D-43 alloy). However, increases in nitrogen content were observed for both the higher nitrogen bearing AS-55 alloy capsules, i.e., from 207 ppm to 236 ppm in the liquid region of capsule #8 and from 203 ppm to 277 ppm in the liquid region of capsule #12. Since capsule #12 was filled with high-purity potassium under vacuum and mass transfer from the condensing region is not evident, the difference in nitrogen content is attributed to inhomogeneity in the sheet and inaccuracies in analyses. Similarly, decreases in the carbon content from 780 ppm to 610 ppm and from 1040 ppm to 850 ppm in the liquid region of AS-55 alloy capsules #8 and #12, respectively, are attributed to inhomogeneity and the limitations in analytical techniques. This is substantiated by 1) the similarity of the post-test carbon analysis of the samples taken from the inner and outer portions of the capsule wall, 2) the fact that the carbon content did not decrease in the liquid and condensing regions of the D-43 alloy capsule #2 and 3) metallographic examination does not reveal a carbide depleted area near the inner surface. No mass transfer of carbon was evident in any of the capsules. Although the hydrogen content appears to have increased in most of the post-test samples, the increase is particularly evident in the samples from capsules #6 (Cb-1Zr alloy) and #8 (AS-55 alloy); this is associated with the fact that both capsules were filled with potassium using the earlier transfer facility. Again, no significance is attached to the increase in hydrogen.

The oxygen contents appear to have increased in both the inner and outer wall portions of essentially all of the capsules as a result of the 5,000-hour exposure with a larger increase in oxygen occurring in the outer wall specimens. The increase in



oxygen in the outside specimens is accounted for mainly by grit blasting with  $\text{Al}_2\text{O}_3$ . A recent investigation<sup>9</sup> has shown that the apparent oxygen contamination from grit blasting with  $\text{Al}_2\text{O}_3$  is severe. A 0.0175-inch thick sheet of Cb-1Zr alloy exhibited an oxygen increase of 842 ppm after grit blasting with  $\text{Al}_2\text{O}_3$ .

In comparing the oxygen analyses obtained from the inner wall specimens, it was found that the oxygen content was always higher in the liquid zone than in the condensing zone, indicating 1) a possible transfer of oxygen from the condensing zone to the liquid zone, 2) removal of the oxygen from the condensing zone by selective leaching by the high-purity condensate and 3) an increase in oxygen content in the liquid zone by a gettering action of the zirconium and yttrium in the columbium. These data are illustrated in Figure 23. From examination of the oxygen analyses of the inner-portion of the capsule walls prior to the 5,000-hour exposure to potassium, it would appear that for the high oxygen-bearing AS-55 alloy, the oxygen was leached from the condenser zone and remained in the liquid potassium as evidenced by the fact that the oxygen was not gettered by the yttrium/zirconium in the AS-55 alloy capsule wall in the liquid zone. It may be hypothesized that the oxygen reacted with yttrium/zirconium taken in solution in the liquid potassium to form finely divided  $\text{Y}_2\text{O}_3$  or a complex yttrium-zirconium oxide. Although the solution of yttrium in potassium has not previously been observed experimentally, yttrium has been shown to be soluble to some extent in a lithium-columbium system at  $1500^\circ\text{F}$ <sup>10</sup>. It also is possible for the oxygen to react with yttrium/zirconium at the surface of the capsule wall in other locations than that which was chemically analyzed. On the other hand, no leaching of the oxygen is apparent in the low oxygen-bearing Cb-1Zr alloy and D-43 alloy capsules, and the increase in oxygen in the inner-capsule wall in the liquid zone is attributed to a gettering action. The high oxygen concentration in the vapor zone of the Cb-1Zr alloy capsule #6 may be the result of the liquid potassium, high in impurities near the liquid-vapor interface, splashing up on the capsule wall. It should be noted that the suspected difference in purity of the potassium used to fill capsule #6 (Cb-1Zr alloy) and #8 (AS-55 alloy) as compared to that used to fill capsules #12 (AS-55 alloy) and #2 (D-43 alloy) does not seem to have an effect on the magnitude of the oxygen gradient observed between the condensing zone and the liquid zone of the capsule after the test exposure.

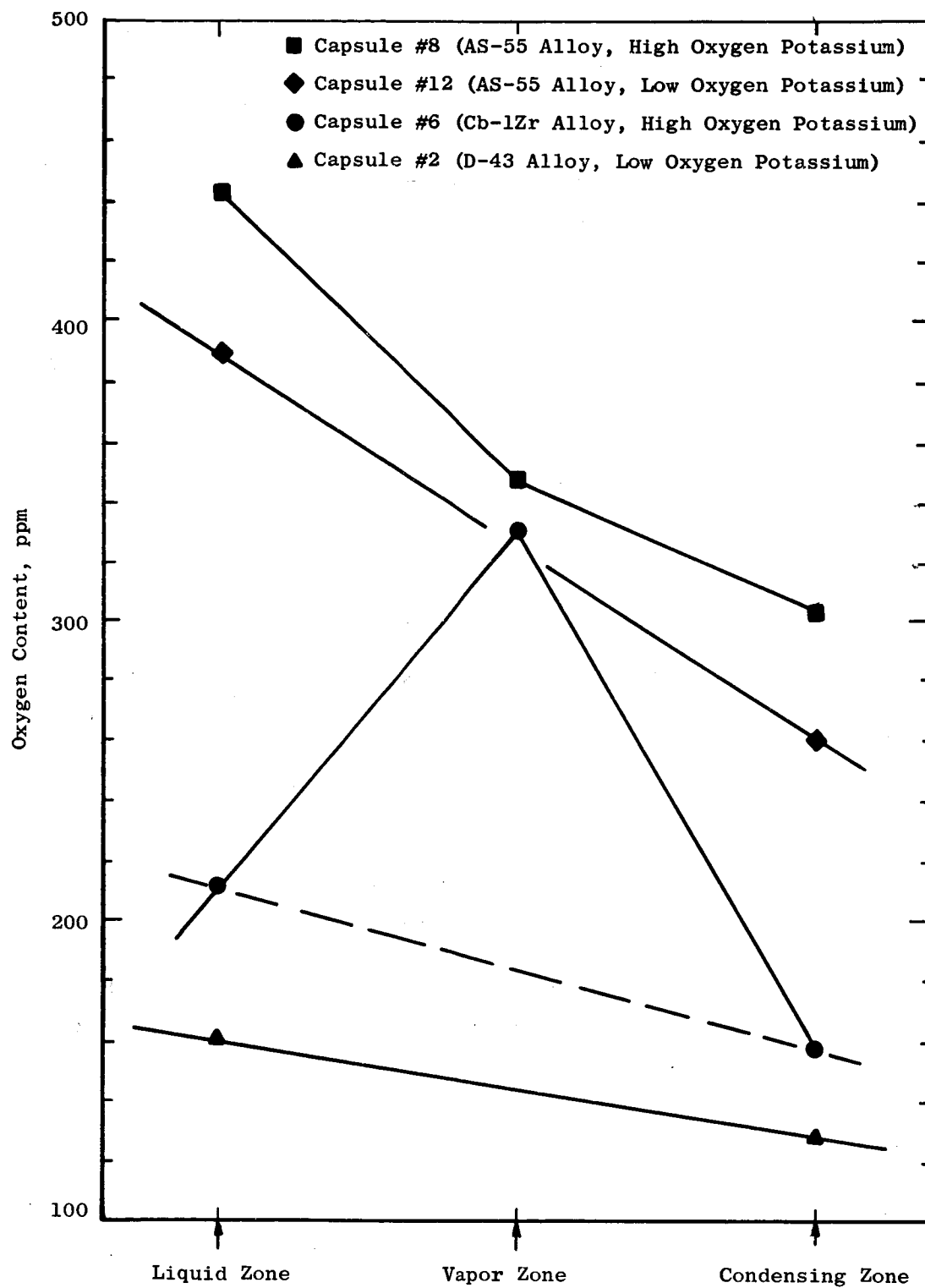


Figure 23. Oxygen Content of Inner 0.040-Inch Layer of Capsule Wall as a Function of Specimen Location. Capsules were Exposed for 5,000 Hours at 2000°F in a Vacuum of  $10^{-9}$  Torr.

## 2. Metallographic Examination

The metallographic examination of the capsules was conducted on samples from the liquid zone, liquid-vapor interface and condensing zone. The locations of the samples are shown in Figure 22.

No significant attack was observed in any of the capsules, Figure 24. The AS-55 alloy capsule #8, that contained potassium which is believed to have had a high concentration of oxygen, showed a slight amount of general solutioning in the liquid zone; this was not evident in the AS-55 alloy capsule #12 that contained potassium of higher purity, Figure 24 (see page 56). A morphological change was observed in the AS-55 alloy. The Widmanstätten structure seen in the matrix of the pre-test AS-55 alloy sheet material has gone into solution and a grain boundary precipitate now exists. Through stain etching techniques, the grain boundary precipitate was tentatively identified as a columbium carbide.

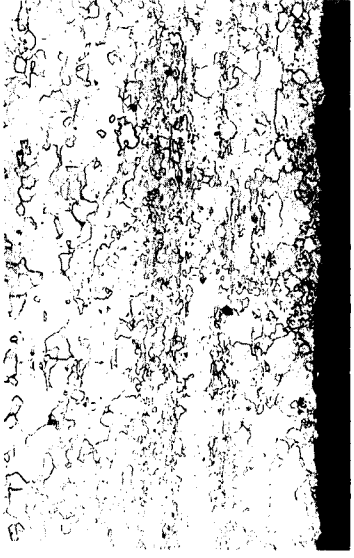
No unusual grain growth during testing was evident in any of the capsules.

In examining the welds of the capsules, again, no significant attack was apparent. Micrographs of the capsule welds in the condensing zones are shown in Figure 25 and are typical of the weld structure observed in the other regions of the capsules. The large grain sizes seen in the weld of the Cb-1Zr and D-43 alloys are quite evident as compared to the smaller grain size of the weld in the AS-55 alloy.

## 3. Hardness Measurements

Transverse microhardness surveys were made across the capsule walls, Figures 26, 27, 28 and 29. The Cb-1Zr alloy capsule #6 showed a slight decrease in hardness after testing. The two high hardness values observed in the pre-test sample of capsule #6 are attributed to a band of residual cold work which was observed metallographically. The AS-55 alloy capsule #8 exhibited a significant decrease in hardness which would follow the carbide solution and reprecipitation previously discussed. Capsule #12, also AS-55 alloy, did not show a hardness decrease. Unfortunately, the pre-test sample for capsule #12 was not heat treated at the same time as the capsule, therefore, resulting in possibly erroneous pre-test data. The D-43 alloy capsule #2 showed a general decrease in hardness which is attributed to recovery during testing since the material was not completely recrystallized prior to testing.

Cb-1Zr Alloy Capsule #6  
High Oxygen Potassium



(L327)

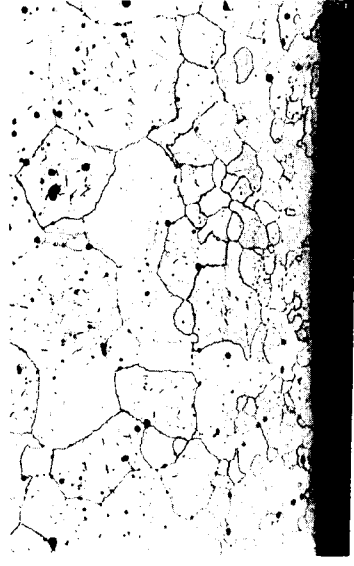
Transverse Section  
of Annealed 0.080-  
Inch Thick Sheet  
Prior to 5,000-Hour  
Exposure

AS-55 Alloy Capsule #8  
High Oxygen Potassium



(K659)

AS-55 Alloy Capsule #12  
Low Oxygen Potassium



(K5414)

D-43 Alloy Capsule #2  
Low Oxygen Potassium



(K5413)

Transverse Section  
of Condensing Zone  
After 5,000-Hour  
Exposure



(K5409)



(K727)

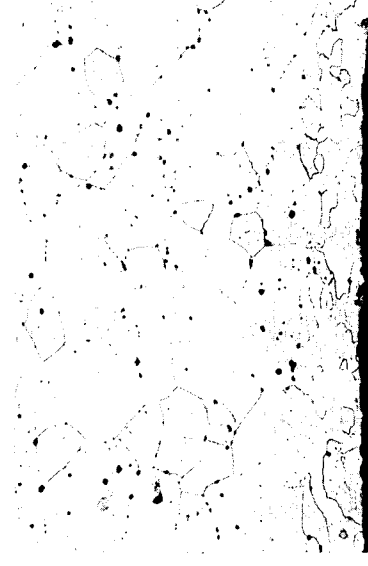
Longitudinal Section  
of Liquid-Vapor Inter-  
face After 5,000-Hour  
Exposure



(L329)



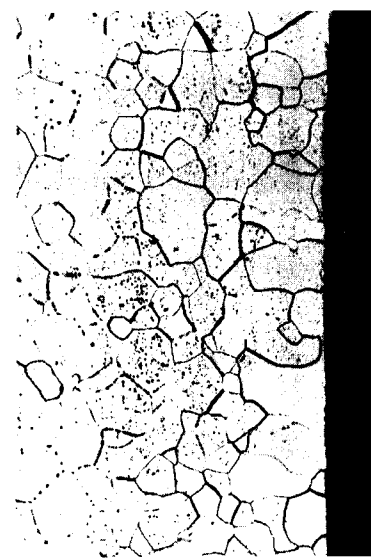
(K5411)



(K3691)



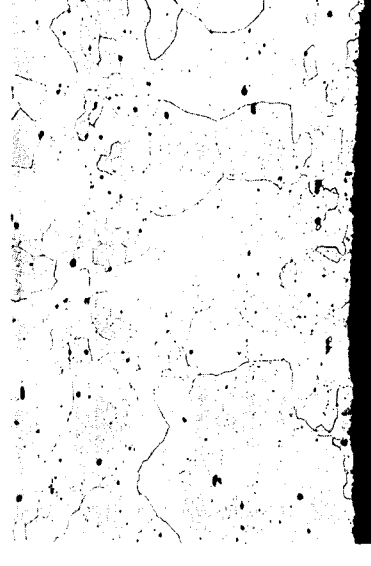
(K817)



(K5408)



(K725)



(K820)



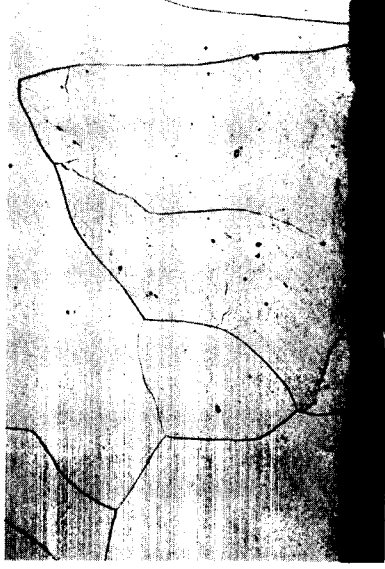
(K815)

(K819)

0 1 2 3 4  
Mils

Figure 24. Microstructures of AS-55, D-43 and Cb-1Zr Alloy Reflux  
Corrosion Capsules Prior to and After 5,000-Hour Expo-  
sure to Potassium at 2000°F in a Vacuum of 10<sup>-9</sup> Torr.  
Etchant: 20%Hf-20%HNO<sub>3</sub>-60%Water Original Mag: 250X

Cb-1Zr Alloy Capsule #6  
High Oxygen Potassium



(K705)

Transverse Section  
of Weld Prior to  
5,000-Hour Exposure

AS-55 Alloy Capsule #8  
High Oxygen Potassium



(K658)

AS-55 Alloy Capsule #12  
Low Oxygen Potassium

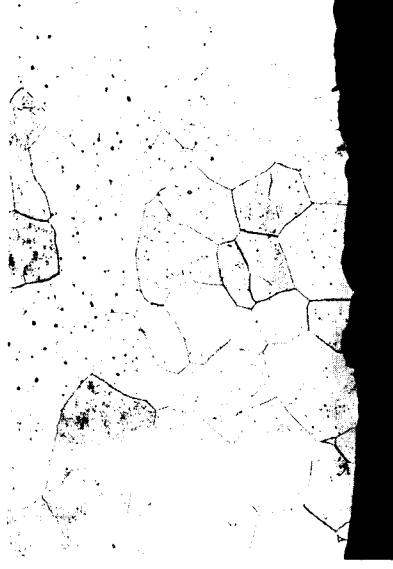


(K726)

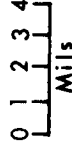


(K5410)

Transverse Section  
of Weld in Condensing  
Zone After 5,000-Hour  
Exposure



(K5412)



D-43 Alloy Capsule #2  
Low Oxygen Potassium



(K816)

Figure 25. Microstructure of Welds in AS-55, D-43 and Cb-1Zr Alloy  
Reflux Corrosion Capsules Prior to and After 5,000-Hour  
Exposure to Potassium at 2000°F in a Vacuum of 10-9 Torr.  
Etchant: 20%Hf-20%HNO<sub>3</sub>-60%Water Original Mag: 250X

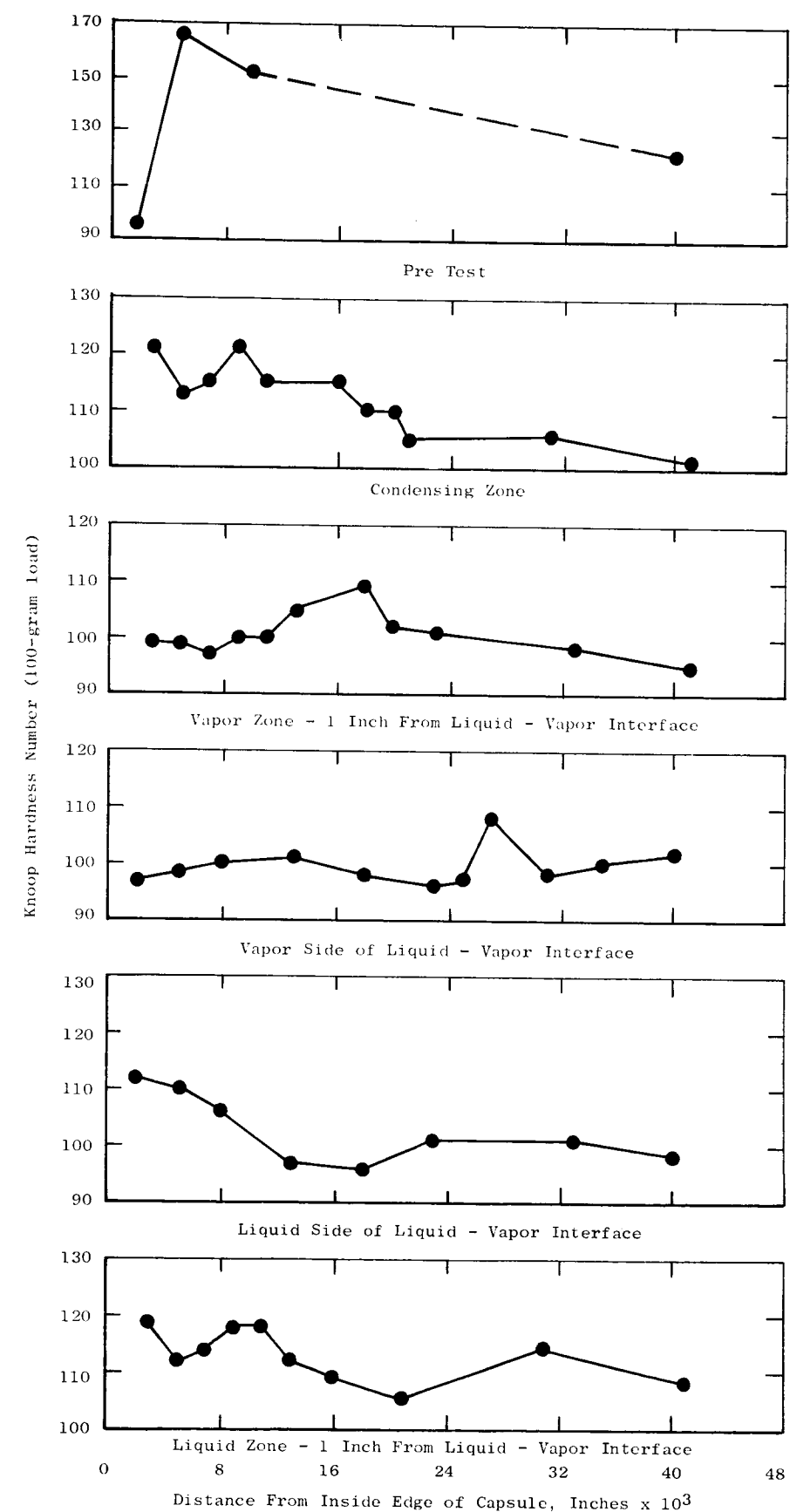


Figure 26. Microhardness Traverses of Transverse Sections of Cb-1Zr Alloy Reflux Corrosion Capsule #6 After 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr.

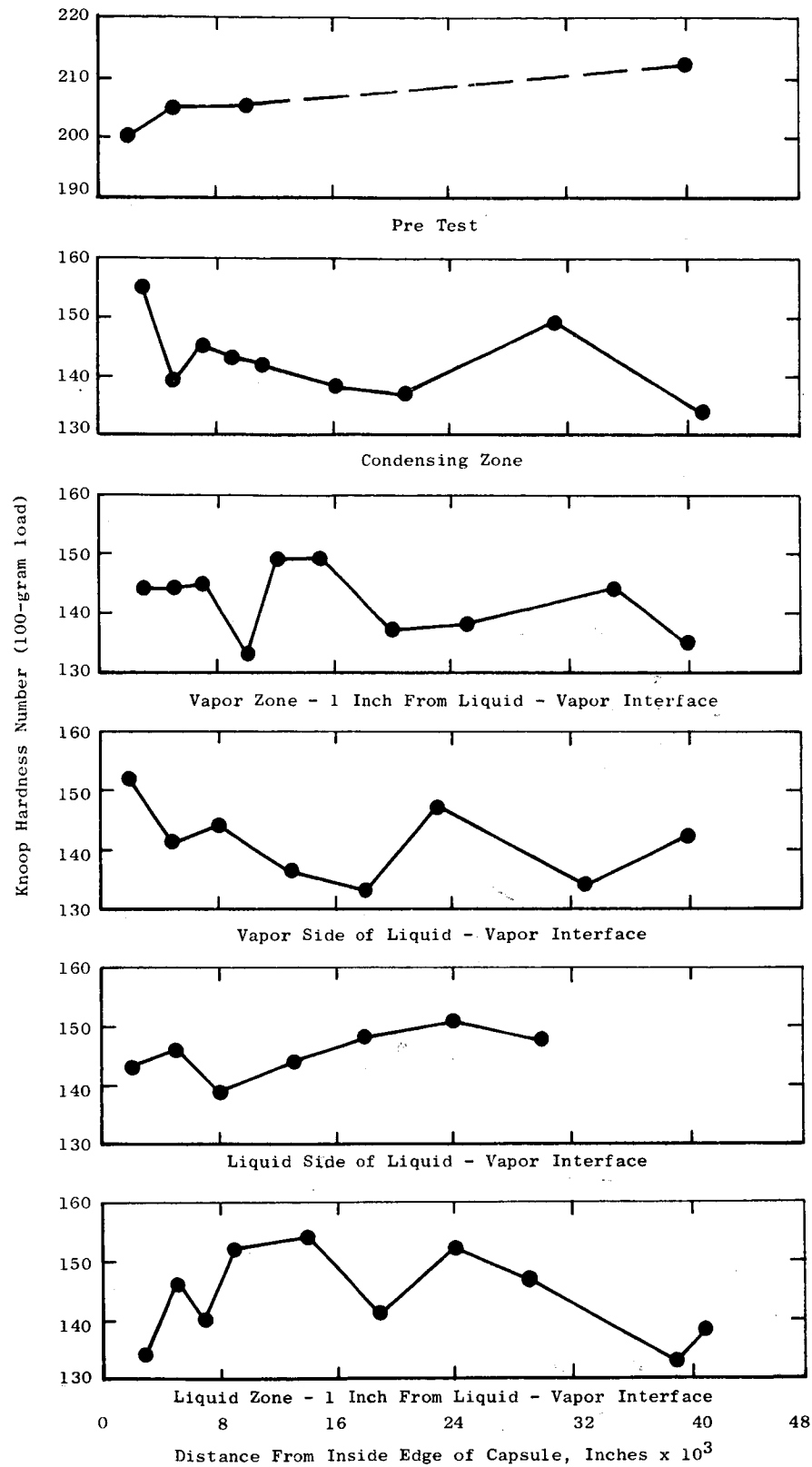


Figure 27. Microhardness Traverses of Transverse Sections of AS-55 Alloy Reflux Corrosion Capsule #8 After 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr.

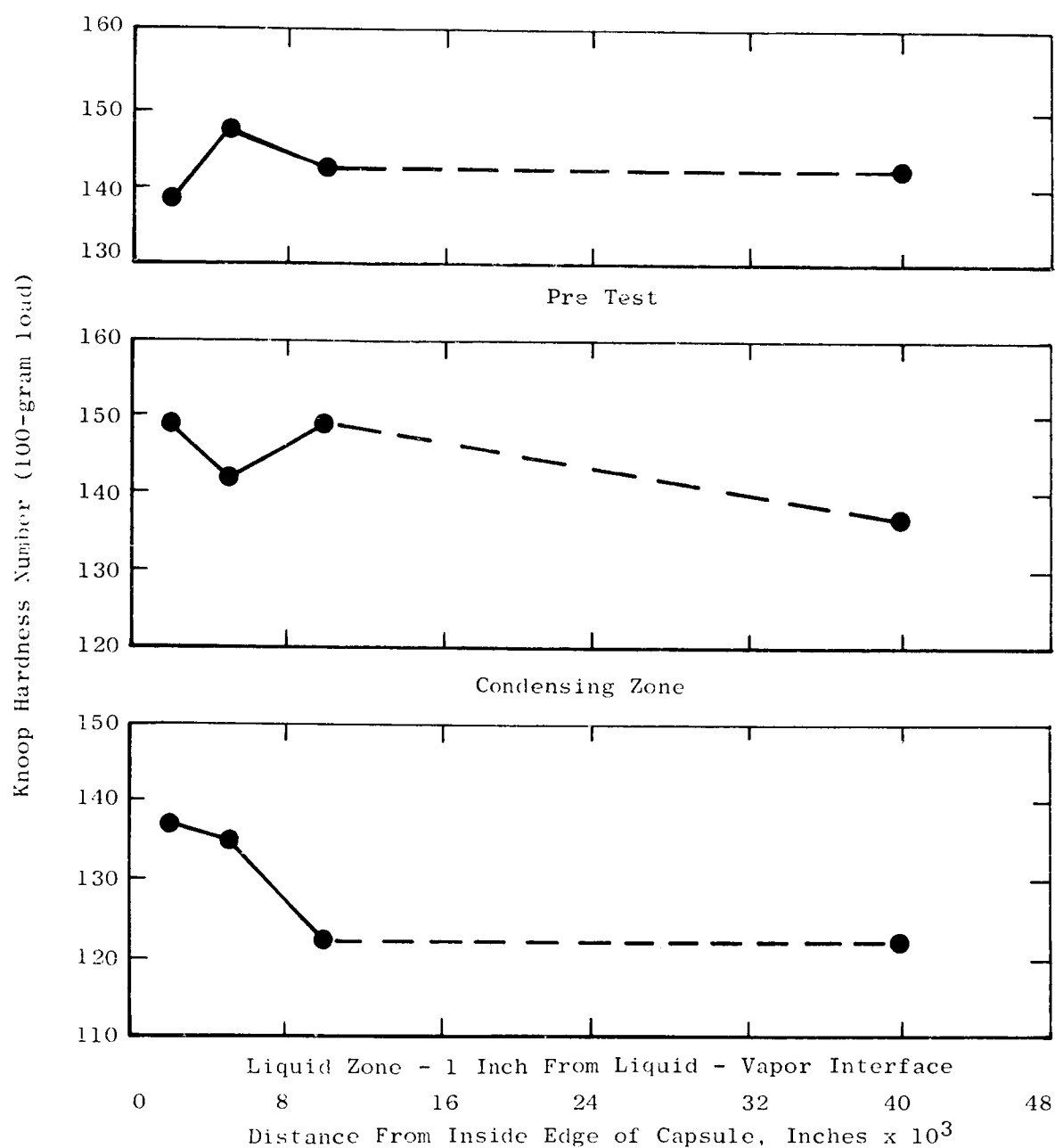


Figure 28. Microhardness Traverses of Transverse Sections of AS-55 Alloy Reflux Corrosion Capsule #12 After 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr.



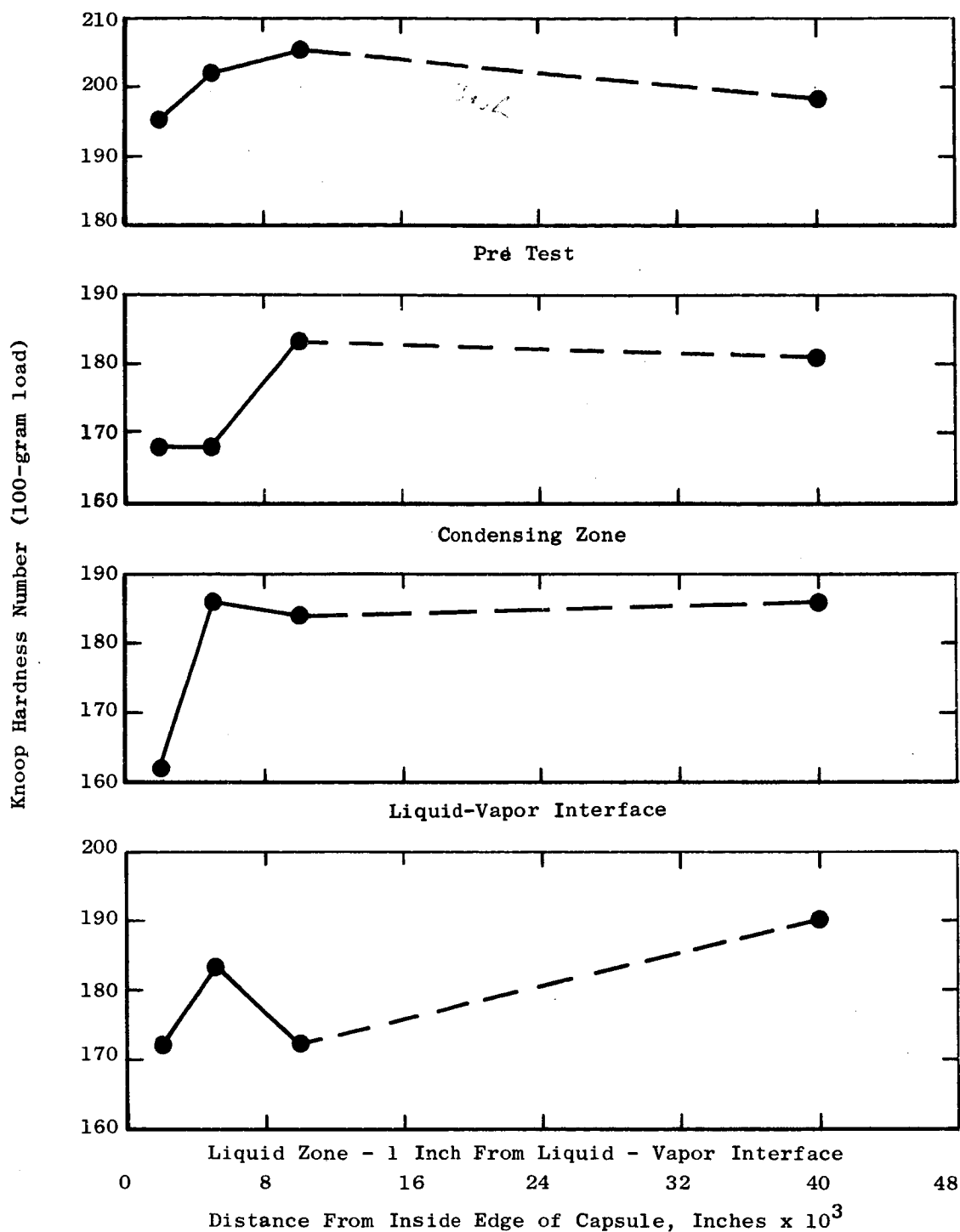


Figure 29. Microhardness Traverses of Transverse Sections of D-43 Alloy Reflux Corrosion Capsule #2 After 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr.

#### 4. Weight Change and Bend Tests

The weight and thickness changes that occurred as a result of subjecting the bend specimens to the potassium environment within the capsules are quite small, and no significance is attached to the data, Table VIII. All of the bend specimens, which were exposed to potassium liquid and vapor, successfully withstood a 1 "T" or smaller bend through a 105° bend angle at room temperature.

#### 5. Stress-Rupture Tests

Two stress-rupture specimens were machined from the wall of the AS-55 capsule #8 and two from the D-43 capsule #2 such that the location of the gauge section of one specimen of each capsule is in the liquid region and the location of the gauge length of the second specimen is in the condensing region. All of the specimens were tested at 2000°F and the results are presented in Table IX with the corresponding Larson-Miller parameter plot shown in Figure 30. The data that were obtained from specimens that had not been exposed to potassium were obtained in the previously described<sup>11</sup> liquid nitrogen trapped, cold-wall oil diffusion pumped, stress-rupture facilities which can achieve a pressure in the 10<sup>-6</sup> torr range at the test temperature. The stress-rupture data that were obtained from specimens that had been exposed to potassium were obtained in a getter-ion pumped (400 Q/sec), high-vacuum stress-rupture facility shown in Figure 31. This facility can achieve pressures in the 10<sup>-9</sup> torr range when cold and in the 10<sup>-8</sup> torr range when testing refractory metals at temperatures on the order of 2000°F. Chemical analyses for the interstitial elements were obtained on a number of specimens after stress-rupture testing to determine the extent of the environmental contamination. The data are presented in Table X.

All specimens which were machined from the wall of the re-flux corrosion capsules after the 5,000-hour exposure to potassium exhibited increased rupture life at 2000°F over the specimens which were machined from the untested material used to fabricate the capsules. Although some coalescence of the carbides appears to have occurred during the 5,000-hour exposure as evidenced by metallographic examination, its influence on strength properties of the AS-55 and D-43 alloys is masked by the change in oxygen concentration at the surface due to gettering action on the inside capsule wall in the case of D-43 alloy, grit blasting of the outside surface of all the capsule

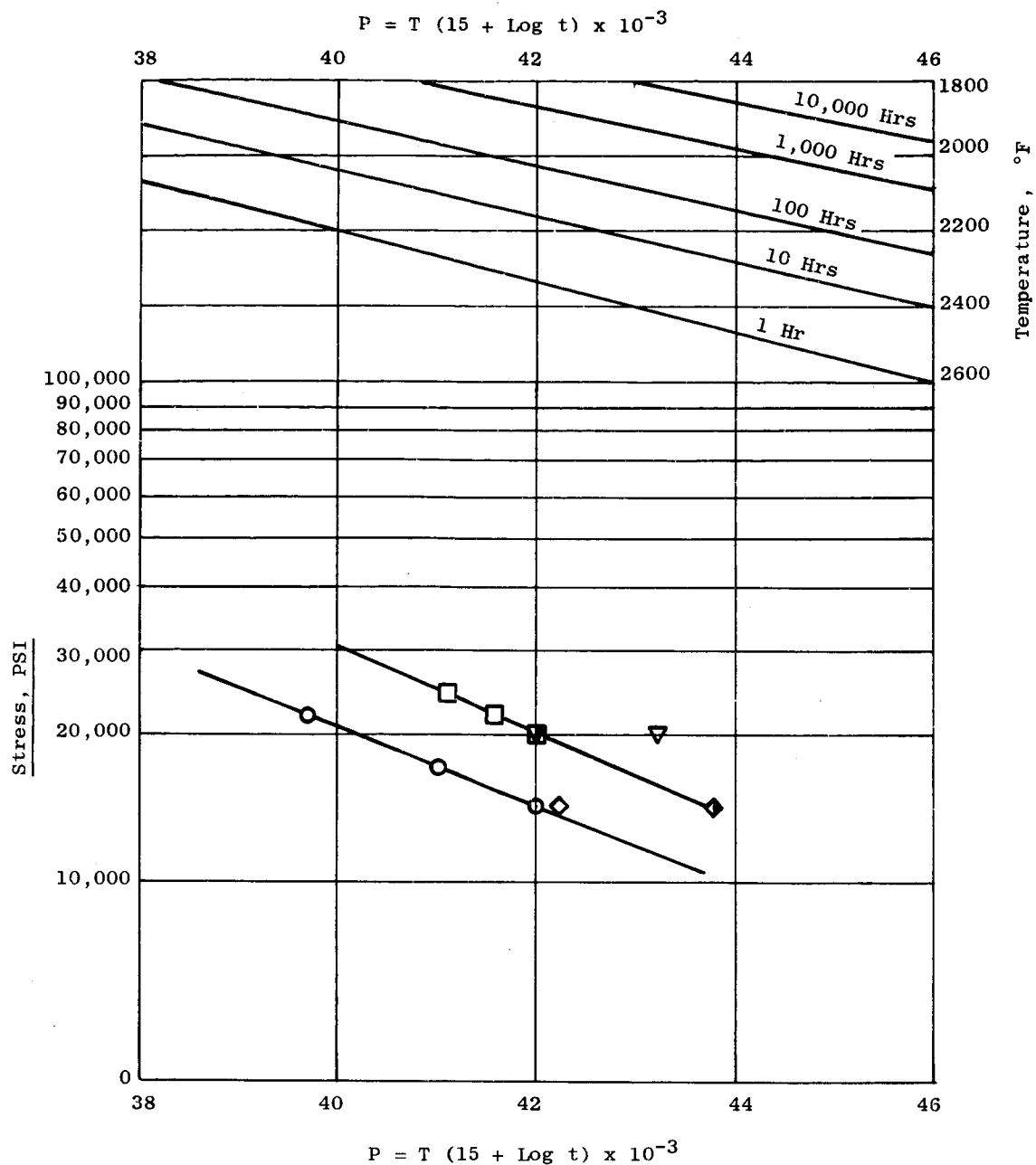
TABLE VIII

THICKNESS AND WEIGHT MEASUREMENTS OF BEND SPECIMENS LOCATED IN REFLUX  
CORROSION CAPSULES AND EXPOSED TO POTASSIUM FOR 5,000 HOURS AT 2000°F

Capsule No.	Thickness(1), Inch		Weight		Weight Change, mg/cm <sup>2</sup>
	Before Exposure	After Exposure	Before Exposure, gm	After Exposure, gm	
#6 (Cb-1Zr) Liquid Vapor	0.0785	0.0782	(2)	(2)	(2)
	0.0840	0.0838	13.2633	13.2635	+0.0002
#8 (AS-55) Liquid Vapor	0.0821	0.0821	(2)	(2)	(2)
	0.0787	0.0782	14.5536	14.5540	+0.0004
#2 (D-43) Liquid Vapor	0.0593	0.0593	7.6049	7.6067	+0.0018
	0.0586	0.0588	8.1196	8.1204	+0.0008
#12 (AS-55) Liquid Vapor	0.0614	0.0617	7.1060	7.1070	+0.0010
	0.0618	0.0620	8.3503	8.3511	+0.0008

(1) Average of Five Measurements Along Specimen Length.

(2) No Data; Specimen was Welded to Bottom of Capsule.



- AS-55 Alloy (NAS-5515), 0.060-Inch Thick Sheet, Heat Treated 1 Hour at 2800°F + 1 Hour at 2400°F, Rupture Tests Performed in Vacuum of  $10^{-6}$  Torr
- ◇ AS-55 Alloy (NAS-5514), Specimen Machined from Wall of Condensing Zone of Reflux Capsule #8, Rupture Test Performed in Vacuum of  $10^{-8}$  Torr
- ◆ AS-55 Alloy (NAS-5514), Specimen Machined from Wall of Liquid Zone of Reflux Capsule #8, Rupture Test Performed in Vacuum of  $10^{-8}$  Torr
- D-43 Alloy (D-43-322), 0.055-Inch Thick Sheet, Heat Treated 1 Hour at 2200°F + 1 Hour at 2400°F, Rupture Tests Performed in Vacuum of  $10^{-6}$  Torr
- ▽ D-43 Alloy (D-43-322), Specimen Machined from Wall of Condensing Zone of Reflux Capsule #2, Rupture Test Performed in Vacuum of  $10^{-8}$  Torr
- ▼ D-43 Alloy (D-43-322), Specimen Machined from Wall of Liquid Zone of Reflux Capsule #2, Rupture Test Performed in Vacuum of  $10^{-8}$  Torr

Figure 30. Stress-Rupture Properties of AS-55 and D-43 Alloy Before and After 5,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr.

TABLE IX

## STRESS-RUPTURE DATA OF AS-55 AND D-43 ALLOYS BEFORE AND

## AFTER 5,000-HOUR EXPOSURE TO POTASSIUM AT 2000°F

Specimen	Test No.	Temp. °F	Stress psi	Pressure Torr	Rupture Life Hours	Elong. in 1 Inch %	Larson-Miller Parameter Value $T(15+\log t) \times 10^{-3}$
AS-55(1) Before Exposure	1	2000	22,000	10 <sup>-6</sup>	14	10.1	39.7
	2	2000	18,000	10 <sup>-6</sup>	43	7.0	41.0
	3	2000	15,000	10 <sup>-6</sup>	113	5.8	42.0
D-43(2) Before Exposure	4	2000	25,000	10 <sup>-6</sup>	51	14.8	41.1
	5	2000	22,000	10 <sup>-6</sup>	89	20.4	41.6
	6	2000	20,000	10 <sup>-6</sup>	118	26.7	42.0
AS-55(3) After 5,000 Hour Exposure Liquid Zone Condensing Zone	7	2000	15,000	10 <sup>-8</sup>	671(5)	1.6	43.8(5)
	8	2000	15,000	10 <sup>-8</sup>	183	19.8	42.4
D-43(4) After 5,000 Hour Exposure Liquid Zone Condensing Zone	9	2000	20,000	10 <sup>-8</sup>	140	28.8	42.1
	10	2000	20,000	10 <sup>-8</sup>	382	30.5	43.2

(1) AS-55 Alloy (NAS-5515); 0.060-Inch Thick Sheet; Heat Treated 1 Hour at 2800°F + 1 Hour at 2400°F. Tests Conducted in Liquid Nitrogen Trapped, Oil Diffusion Pumped System.

(2) D-43 Alloy (D-43-322); 0.055-Inch Thick Sheet, Heat Treated 1 Hour at 2200°F + 1 Hour at 2400°F. Tests Conducted in Liquid Nitrogen Trapped. Oil Diffusion Pumped System.

(3) AS-55 Alloy (NAS-5514); Specimen Machined from Wall of Reflux Capsule #8. Tests Conducted in Getter-Ion Pumped System.

(4) D-43 Alloy (D-43-322); Specimen Machined from Wall of Reflux Capsule #2. Tests Conducted in Getter-Ion Pumped System.

(5) Specimen Did Not Fail.

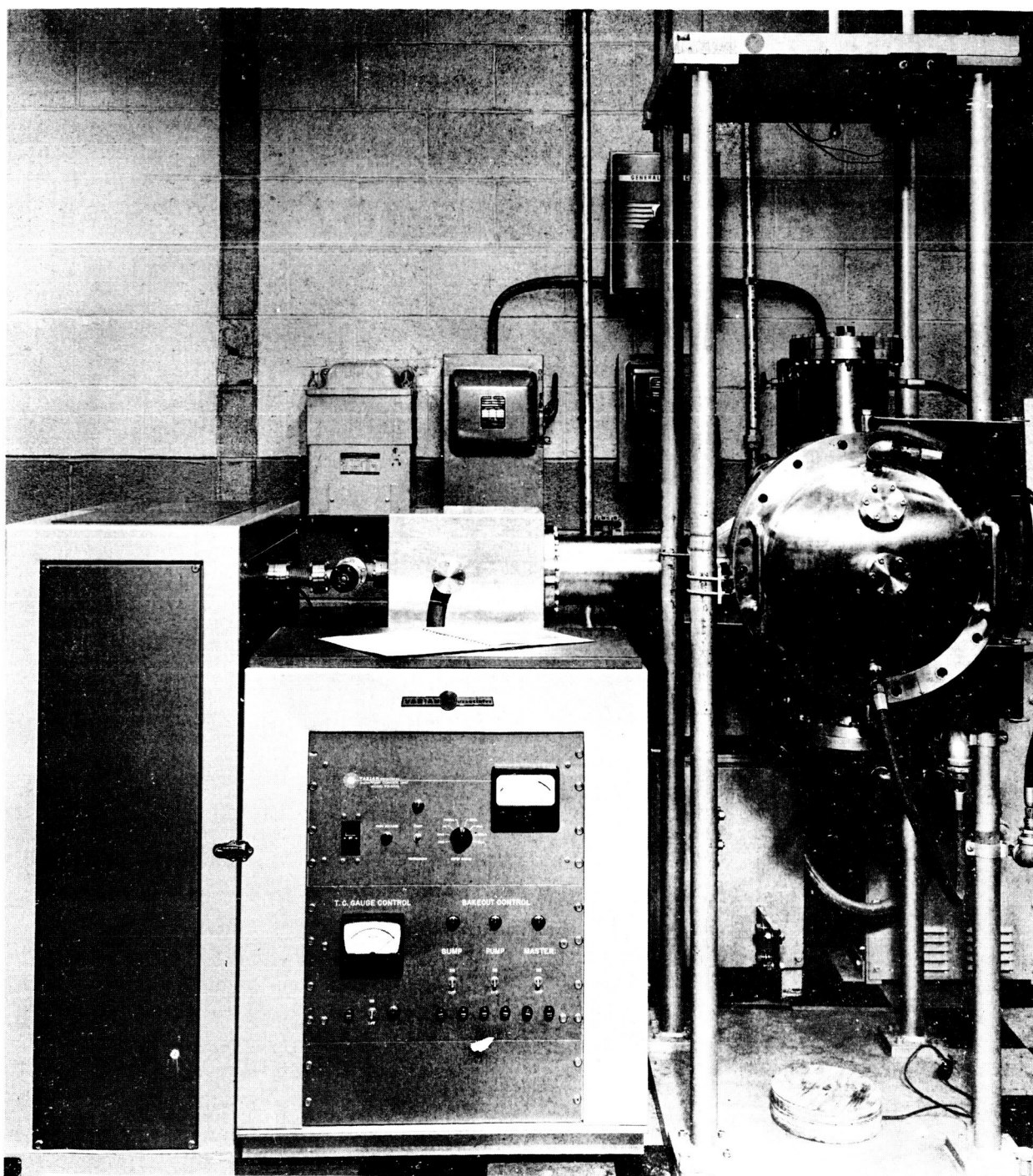


Figure 31. High-Vacuum Stress-Rupture Facility Equipped With a 400  $\text{L}/\text{Sec}$  Getter-Ion Pump and Capable of Achieving a Pressure of  $10^{-9}$  Torr. (C63020501)

TABLE X

CHEMICAL ANALYSES (w/o) OF AS-55 AND D-43 ALLOY BEFORE  
AND AFTER STRESS-RUPTURE TESTING AT 2000°F IN VACUUM

Specimen	Test No.	Rupture Life Hours	Pressure Torr	Before Test			After Test				
				<u>O</u>	<u>N</u>	<u>H</u>	<u>C</u>	<u>O</u>	<u>N</u>	<u>H</u>	<u>C</u>
AS-55(1)	3	113	10 <sup>-6</sup>	0.0356	0.0192	0.0002	0.106	0.0324	0.0213	<0.0001	0.065
D-43(2)	6	118	10 <sup>-6</sup>	0.0110	0.0026	0.0004	0.101	0.0076	0.0023	<0.0001	0.084
AS-55(3) Liquid Zone	7	671(5)	10 <sup>-8</sup>	0.0498	0.0204	0.0008	0.060	0.0646	0.0237	0.0003	---
D-43(4) Condensing Zone	10	382	10 <sup>-8</sup>	0.0138	0.0027	0.0004	0.108	0.0221	0.0050	0.0001	---

(1) AS-55 Alloy (NAS-5515); 0.060-Inch Thick Sheet; Heat Treated 1 Hour at 2800°F + 1 Hour at 2400°F. Test Conducted in Liquid Nitrogen Trapped, Oil Diffusion Pumped System.

(2) D-43 Alloy (D-43-322); 0.055-Inch Thick Sheet; Heat Treated 1 Hour at 2200°F + 1 Hour at 2400°F. Test Conducted in Liquid Nitrogen Trapped, Oil Diffusion Pumped System.

(3) AS-55 Alloy (NAS-5514); Specimen Machined from Wall of Reflux Capsule #8. Test Conducted in Getter-Ion Pumped System.

(4) D-43 Alloy (D-43-322); Specimen Machined from Wall of Reflux Capsule #2. Test Conducted in Getter-Ion Pumped System.

(5) Specimen Did Not Fail.

walls and possible contamination during stress-rupture testing. Additional tests in progress on specimens machined from the wall of the duplicate AS-55 alloy reflux corrosion capsule which was exposed to potassium from 5,000 hours are expected to provide a greater insight as to the effect of a long time exposure to boiling potassium at elevated temperature on the strength properties of AS-55 alloy. In these tests, the outer surface of the specimens has been removed and the tests are being conducted in an improved vacuum system.

#### E. Status of 10,000-Hour Reflux Capsule Corrosion Tests

One Cb-1Zr alloy capsule, #7, two AS-55 alloy capsules, #9 and #11, and one D-43 alloy capsule, #4, containing purified potassium, have completed 10,000 hours of testing at 2000°F under refluxing conditions.

Visual examination of the Cb-1Zr alloy capsule #7 revealed discolorations in the region between the primary condensing zone and the liquid zone, Figure 32. Black deposits, accompanied by a gold discoloration of the metal around or near the deposits, also were found in this region, Figure 33. In addition, a change in the general surface appearance of the heat-affected zone of the weld and a more lustrous appearance of the surface above the liquid zone was noted.

The AS-55 alloy capsule #9 also showed discolorations in the region between the primary condensing zone and the liquid zone, but to a much lesser degree than those found in the Cb-1Zr alloy capsule #7, Figure 34. A dark grey spot surrounded by a white deposit was noted on the inside wall in the liquid region opposite the location where a thermocouple was attached to the outer wall. Also, as in capsule #7 (Cb-1Zr alloy), a change in the general surface appearance in the heat-affected zone of the weld and a more lustrous appearance above the liquid zone were observed.

AS-55 alloy capsule #11, which was filled with potassium using the vacuum filling facility discussed earlier, showed no discolorations in the region just above the liquid zone, Figure 35. However, white deposits, which were similar to those seen previously in the 5,000-hour AS-55 alloy capsule #12, were found in three locations on the inside surface of the AS-55 capsule #11; two in the liquid region and one in the vapor region. Figure 36 shows the white deposits that formed on the weld in the liquid zone and Figure 37 shows a deposit on the parent metal in the liquid region opposite



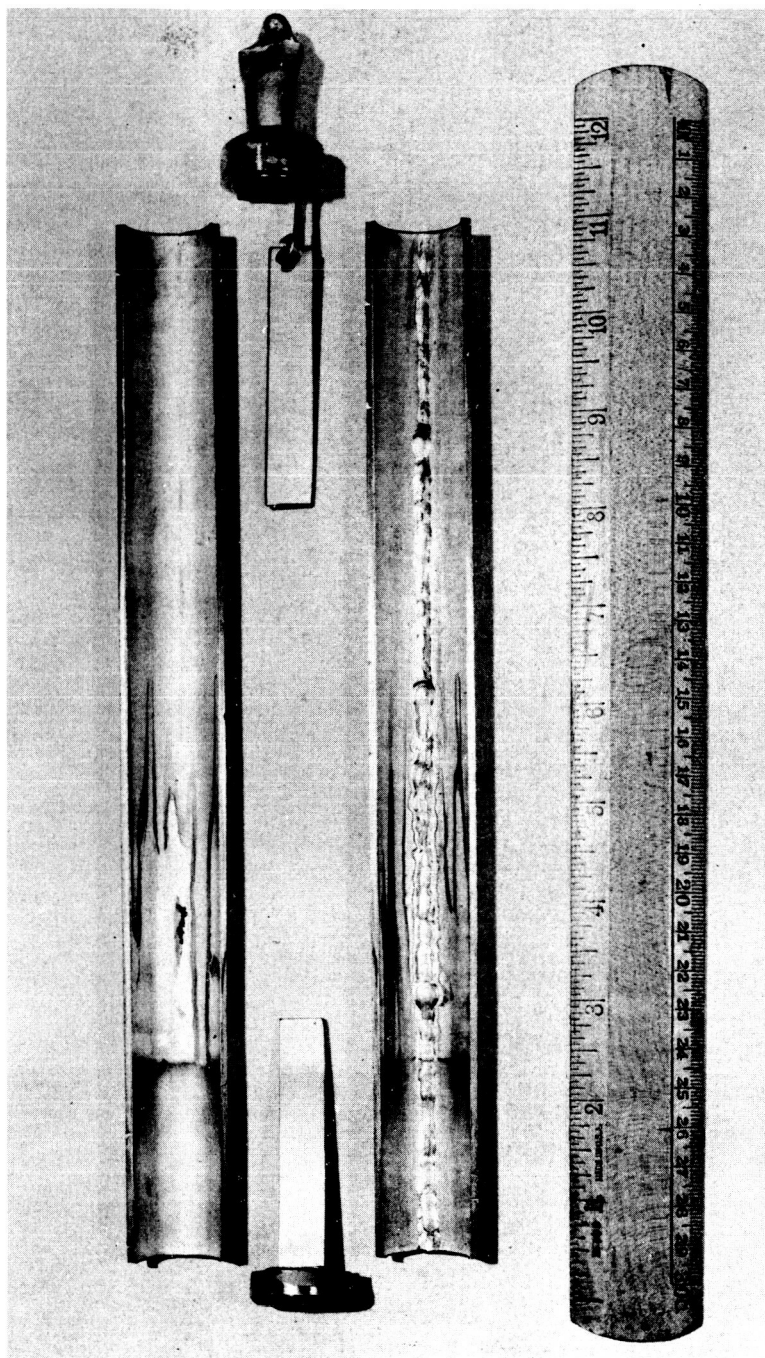


Figure 32. Sectioned Cb-1Zr Alloy Reflux Corrosion Capsule #7  
After a 10,000-Hour Exposure to Potassium at 2000°F  
in a Vacuum of  $10^{-9}$  Torr. (C63122633)



Figure 33. Black Deposits and Discoloration Observed on the Inner Surface of Cb-1Zr Alloy Corrosion Capsule #7 Between the Primary Condensing Zone and the Liquid Zone After a 10,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr.

Mag: 2X (C63122625)

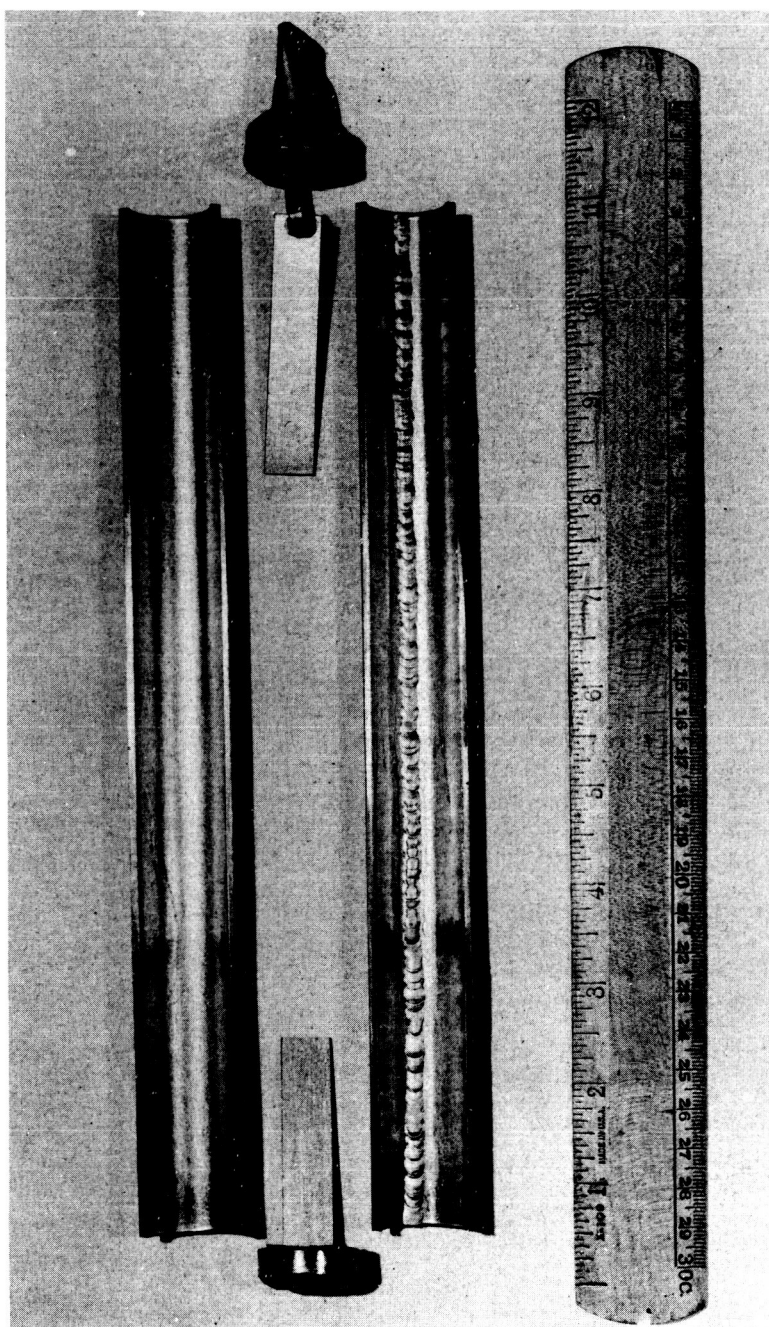


Figure 34. Sectioned AS-55 Alloy Reflux Corrosion Capsule #9  
After a 10,000-Hour Exposure to Potassium at 2000°F  
in a Vacuum of  $10^{-9}$  Torr. (C63122632)

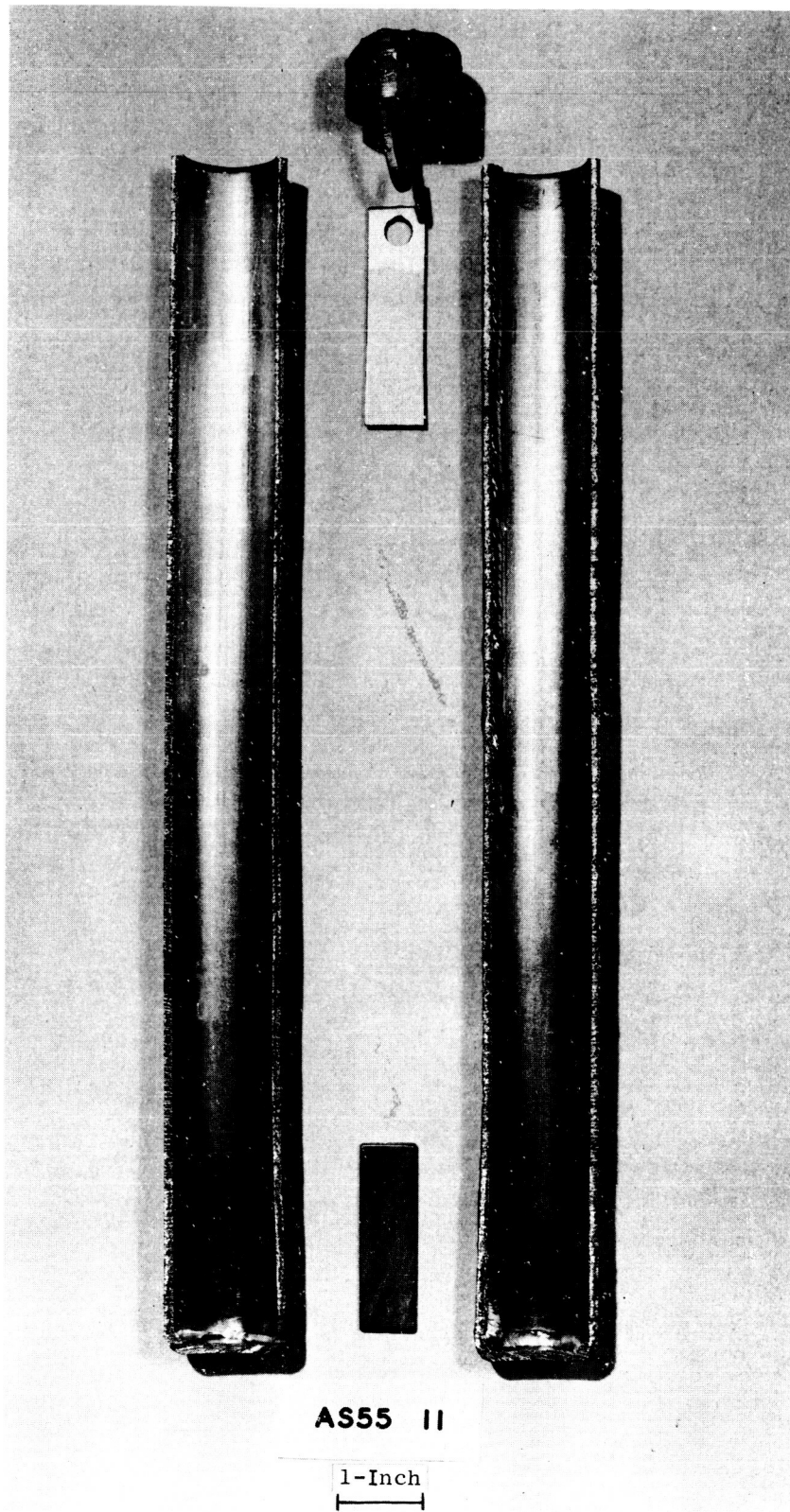


Figure 35. Sectioned AS-55 Alloy Reflux Corrosion Capsule #11  
After a 10,000-Hour Exposure to Potassium at 2000°F  
in a Vacuum of  $10^{-9}$  Torr. (C64091109),

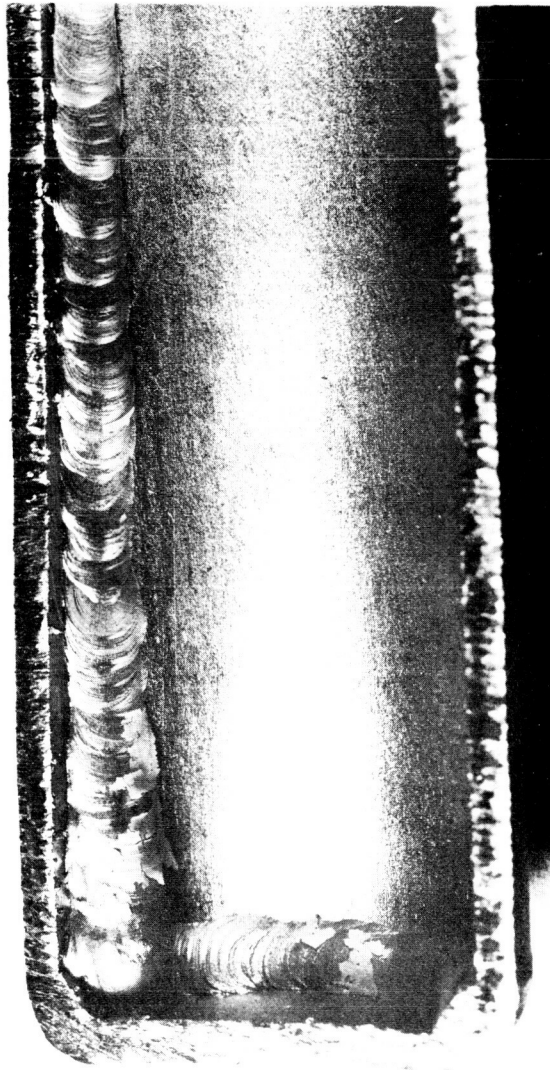


Figure 36. White Deposit Found in Weld in Liquid Zone of AS-55 Alloy Reflux Capsule #11 After a 10,000-Hour Exposure to Potassium at 2000°F in a Vacuum of  $10^{-9}$  Torr.  
Mag: 3X (C64091112)

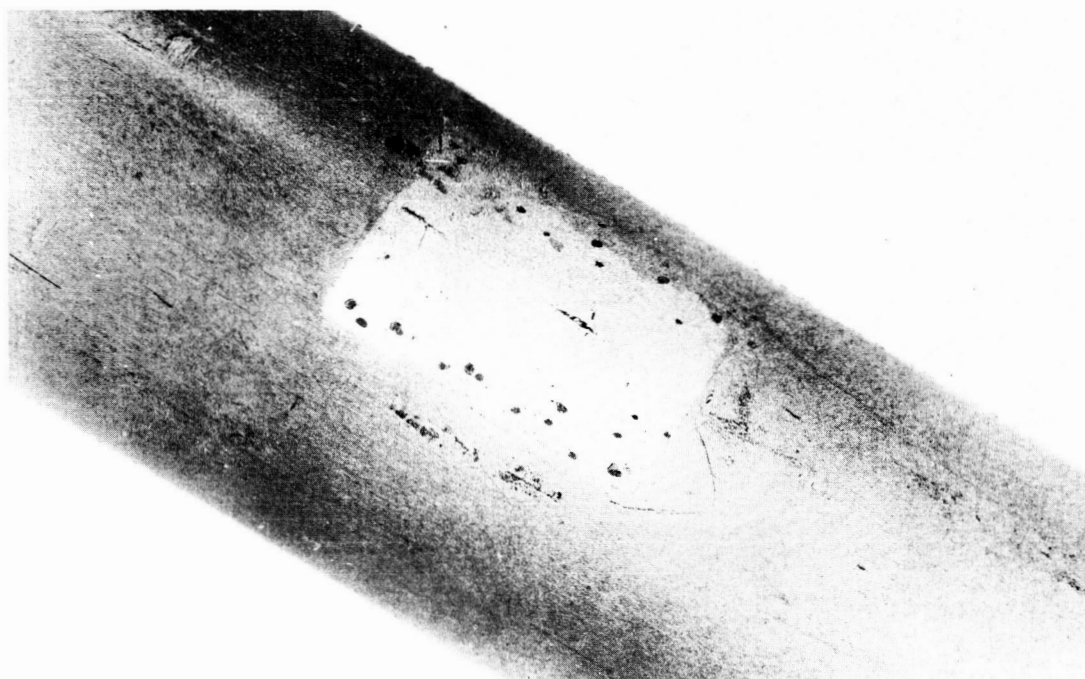
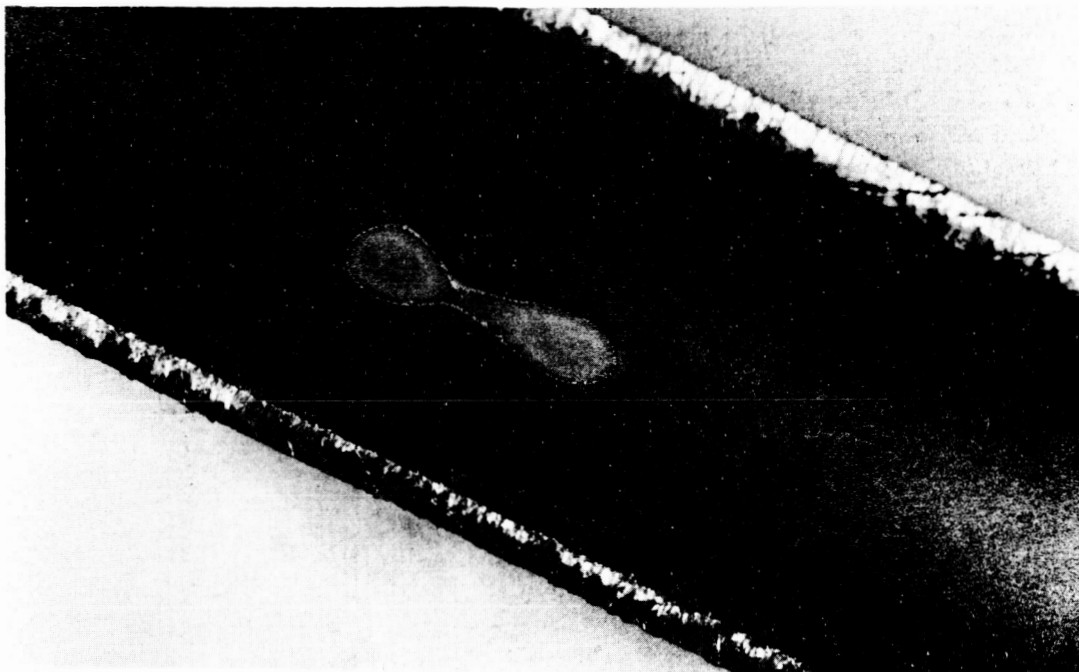


Figure 37. White Deposits Found on the Inside Surface of AS-55 Alloy Reflux Capsule #11 in the Liquid Zone (Top) Opposite the Location Where a Thermocouple was Attached to the Capsule Wall (Bottom). The Capsule was Exposed to Potassium for 10,000-Hours at 2000°F in a Vacuum of  $10^{-9}$  Torr. Note Bonding of Tantalum Sheath of Thermocouple to Capsule. Top: Mag: 3X (C64031113); Bottom: Mag: 3X (C64031111).



the point where a thermocouple was in contact with the capsule wall. Although the majority of the deposited material was found in the liquid region, one small deposit was found in the vapor region. In previous capsules, i.e., 5,000-hour tests, similar deposits have been observed only in the liquid zone. Identification of the deposit in capsule #11 is currently under way. However, it is postulated that the deposit is  $Y_2O_3$  which is formed by the oxidation of yttrium in the potassium and on the surface of the capsule wall. The primary mechanism which is believed to account for the presence of  $Y_2O_3$  at localized areas on the capsule wall assumes the existence of localized areas in the capsule wall that can provide a relatively large source of oxygen, resulting in the oxidation of yttrium that is in solution with the potassium at these specific locations. In the case of the weld metal, a high oxygen concentration could exist due to contamination during the welding operation. The presence of a high oxygen concentration in localized spots in the parent metal opposite thermocouple junctions is most likely due to diffusion of oxygen from the thermocouple insulation resulting from non-stoichiometry, entrapment of air during swagging or from impurities in the  $Al_2O_3$ . Because of the latter possibilities, the use of bare thermocouple wires is recommended.

Evidence to support the belief that oxygen from the  $Al_2O_3$  thermocouple insulation diffused through the tantalum sheath and capsule wall to react with the yttrium at the potassium/capsule interface, Figure 36, was obtained from metallographic examination and microhardness traverses of the capsule wall in the area where the tantalum-sheathed thermocouple had bonded to the capsule wall.

A second mechanism that could play a part in the formation of  $Y_2O_3$  at localized areas is the mass transport of yttrium due to a small localized temperature gradient and the subsequent oxidation of the yttrium to  $Y_2O_3$  by reaction with oxygen in the potassium and/or the capsule material. Since the location of the deposits was either at the bottom corner of the capsules or corresponded to the location where the tantalum-sheathed thermocouples had bonded to the outer capsule wall, it is possible that heat could be extracted from the capsule wall at these locations by conduction through the capsule support at the bottom and by conduction along the tantalum sheath, resulting in "cold spots".

Both of the above mechanisms assume some solubility of yttrium in potassium. As discussed previously, although the solution of yttrium in potassium has not been observed experimentally, yttrium

has been shown to be soluble to some extent in a lithium-columbium system at 1500°F<sup>10</sup>. This subject will be covered further in the forthcoming report covering the results of the 10,000-hour capsule tests.

After sectioning, the D-43 alloy capsule #4 revealed a lustrous appearance throughout with the liquid-vapor interface and the vapor-condenser interface being more discernible than could be observed in the 5,000-hour D-43 alloy capsule #2, Figure 38.

Overall, the 10,000-hour exposures to potassium at 2000°F have resulted in negligible corrosion and the more subtle effects of the exposure are being evaluated in a manner similar to that described for the 5,000-hour capsules. It is very probable that similar results would be obtained on any columbium or tantalum base alloy containing strong oxide formers such as yttrium, zirconium or hafnium.



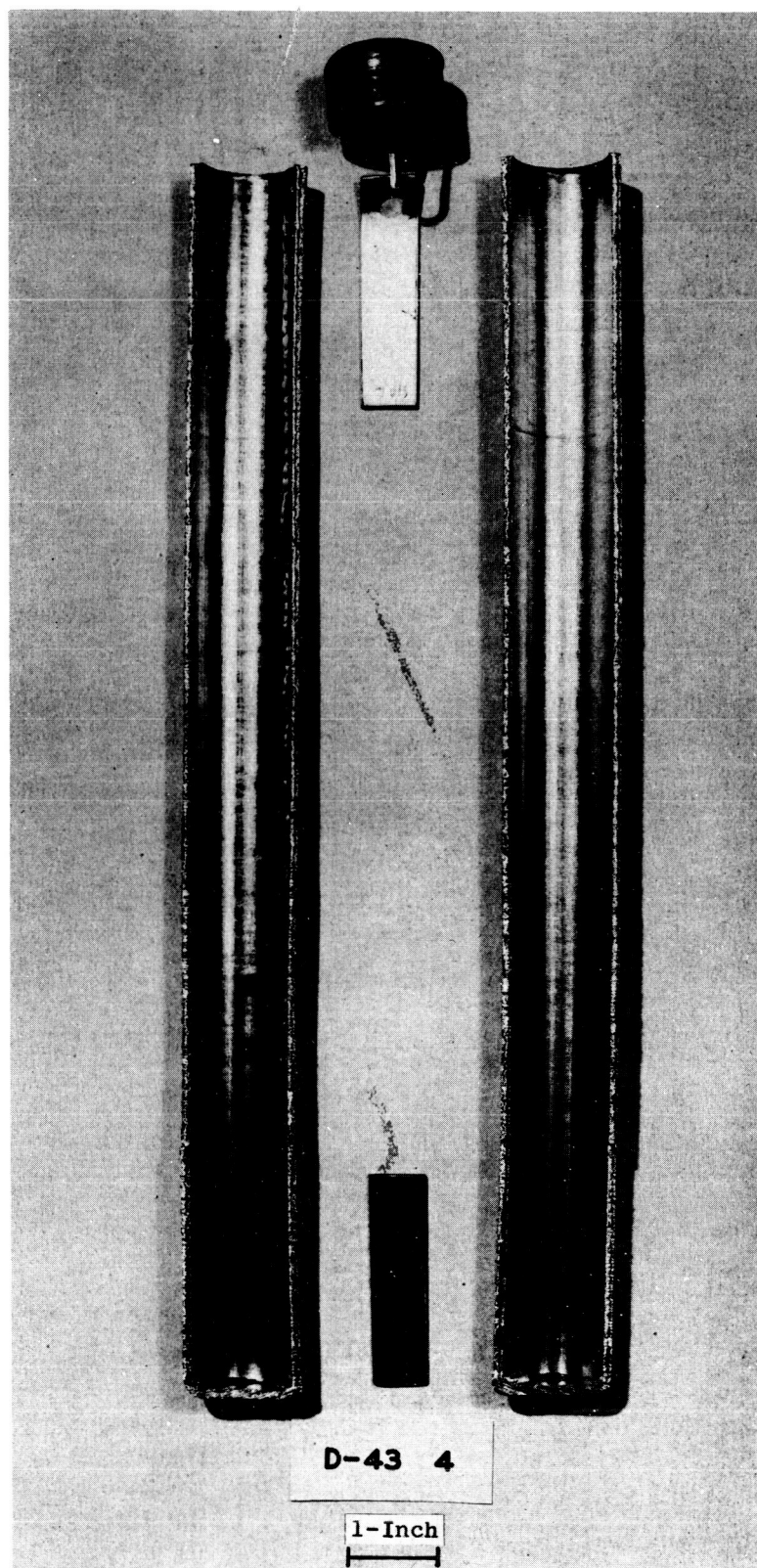


Figure 38. Sectioned D-43 Alloy Reflux Corrosion Capsule #4  
After a 10,000-Hour Exposure to Potassium at 2000°F  
in a Vacuum of  $10^{-9}$  Torr. (C64091110)

#### IV. APPENDIX

APPENDIX A

SUMMARY OF PROCESSING DETAILS OF AS-55, D-43 AND Cb-1Zr  
ALLOY SHEET USED IN THE FABRICATION OF REFLUX CORROSION CAPSULES

SUMMARY OF THE PROCESSING OF AS-55 ALLOY HEAT NAS-555

1. Single consumable D.C. arc melt.
2. Cropped ingot dimensions: 2.9 inches diameter x 4.45 inches long - 10.75 pounds.
3. Ingot split lengthwise and one-half machined into forging billet.
4. Forging billet dimensions: 1.22 inches thick x 1.75 inches wide x 4.30 inches long - 2.87 pounds.
5. Titanium clad forging billet dimensions: 1.333 inches thick x 3.93 inches wide x 6.43 inches long.
6. Clad billet soaked 45 minutes at 2200°F in argon.
7. Five blows on 2,500-pound forge hammer to 0.800-inch thick over-all - 39% reduction.
8. Clad forging re-soaked 15 minutes at 2200°F in argon.
9. Clad forging rolled in the direction of the long axis of the ingot:

<u>Pass</u>	<u>Thickness, In.</u>	<u>% Red./Pass</u>	<u>% Red., Total</u>
1	0.613	24	24
2	0.465	24	42
3	0.322	30	60

Soaked 15 minutes at 2200°F between passes.  
Plate turned end for end each pass.

10. Stripped, pickled and trimmed - plate dimensions - 0.264 inches thick x 3.10 inches wide x 10.70 inches long - 2.7 pounds.
11. One-third of plate sectioned and discarded (low stirring region of original ingot).
12. Plate cold rolled 65% to 0.080-inch thick sheet by successive 10-mil reductions.
13. Sheet stress-relieved one hour at 2300°F.

SUMMARY OF THE PROCESSING OF AS-55 ALLOY HEAT NAS-5514

1. Single consumable D.C. arc melt.
2. Cropped ingot dimensions: 3.05 inches diameter x 4.37 inches long - 11.31 pounds.
3. Ingot split lengthwise and one half-machined into forging billet.
4. Forging billet dimensions: 1.00 inch thick x 1.77 inches wide x 3.93 inches long - 2.25 pounds.
5. Titanium clad billet dimensions: 1.124 inches thick x 4.37 inches wide x 6.50 inches long.
6. Clad billet soaked 45 minutes at 2200°F in argon.
7. Six blows on 2,500-pound forge hammer to 0.550-inch thick over-all - 51% reduction.
8. Clad forging re-soaked 15 minutes at 2200°F in argon.
9. Clad forging rolled in direction of the long axis of the ingot on first pass, normal to the long axis on the second pass, and returned to the original direction on the final pass.

<u>Pass</u>	<u>Thickness, In.</u>	<u>% Red./Pass</u>	<u>% Red., Total</u>
1	0.443	19	19
2	0.353	20	35
3	0.280	20	49

Soaked 15 minutes at 2200°F between passes.  
Plate turned end for end each pass.

10. Stripped, pickled and trimmed - plate dimensions: 0.216 inches thick x 3.87 inches wide x 6.5 inches long - 1.68 pounds.
11. Plate cold rolled 62% to 0.080-inch thick in successive 10-mil reduction.
12. Sheet stress-relieved one hour at 2300°F.

SUMMARY OF THE PROCESSING OF AS-55 ALLOY HEAT NAS-5515

1. Single consumable D.C. arc melt.
2. Cropped ingot dimensions: 3.65 inches diameter x 10.3 inches long - 36.9 pounds.
3. Ingot sectioned to remove defective area.
4. Billet sealed in molybdenum can.
5. Clad billet extruded 5.6:1 at 2600°F to 0.70-inch thick x 2.68 inches wide sheet bar.
6. Clad extrusion soaked 45 minutes at 2200°F in argon.
7. Clad extrusion rolled in direction perpendicular to extrusion direction:

<u>Pass</u>	<u>Thickness, In.</u>	<u>% Red./ Pass</u>	<u>% Red., Total</u>
1	0.632	10	10
2	0.546	13	22
3	0.454	16	35
4	0.360	20	48

Soaked 15 minutes at 2200°F between passes.  
Plate turned end for end each pass.

8. Clad plate stripped, pickled, spot ground and trimmed.
9. Plate cold rolled 75% to 0.082-inch thick sheet by successive 0.010-inch reductions.
10. Sheet stress-relieved one hour at 2200°F.

SUMMARY OF THE PROCESSING OF D-43 ALLOY HEAT D-43-322

1. Double consumable D.C. arc melt.
2. Ingot dimensions: 8 inches diameter.
3. Billet extruded 4:1 at 2000°F to 2 inches thick x 6 inches wide sheet bar.
4. Extrusion warm rolled at 2000°F - reduced temperature gradually until sheet was 0.250-inch thick.
5. Plate annealed one hour at 2200°F.
6. Plate cold rolled to 0.130-inch thick sheet.
7. Sheet annealed one hour at 2200°F.
8. Sheet cold rolled to 0.080-inch thick sheet.
9. Sheet stress-relieved one hour at 2200°F.

SUMMARY OF THE PROCESSING OF Cb-1Zr ALLOY HEAT 519

1. Single electron beam melt.
2. Ingot dimensions: 5 inches diameter x 20 inches long - 120 pounds.
3. Ingot warm forged at 400°F to plate: 1.125 inches thick x 12 inches wide x 20 inches long.
4. Plate cold rolled to 0.100-inch thick sheet.
5. Sheet annealed one hour at 2200°F.



APPENDIX B

DATA SHEETS FOR THE CHEMICAL ANALYSES OF  
POTASSIUM BY THE ZIRCONIUM-GETTER TECHNIQUE

TABLE I

## POTASSIUM ANALYSES BY THE ZIRCONIUM-GETTERING TECHNIQUE

Auxiliary Capsule No.	Zirconium Sample No.	Zirconium Analyses, ppm				Potassium Impurities, ppm (Calculated)				Remarks
		O	N		C	O	C			
			H	H			H	C		
2	a	1740	17	25	-	712	-	-	K sampled during filling of Cb-1Zr alloy capsules #6 and #7 and AS-55 alloy capsules #8 and #9	
	b	1430	16	7	-	502	-	-		
	c	2320	20	31	-	1104	-	-		
	d	1760	19	54	300	725	133			
	Avg.	1813	18	29	300	760	133			

NOTES: (1) Auxiliary capsule exposed in a vacuum at 1400°F for 100 hours.

(2) Cb-1Zr alloy reaction capsule 1-inch diameter x 6-inch long x 0.080-inch thick wall.

(3) Initial analysis of zirconium, ppm:

Sample	O	N	H	C
a	638	19	18	-
b	840	14	25	70
c	587	31	40	135
Avg.	688	21	28	103

(4) Auxiliary capsule #2 contained 7.14 gm. of potassium, 4.828 gm of zirconium (20 square inch surface area).

(5) Analysis of oxygen, nitrogen and hydrogen in zirconium by vacuum fusion techniques.

(6) Analysis of carbon in zirconium by conductometric techniques.

(7) Potassium from shipping container A-4 - slugged, filtered, distilled and hot trapped.

TABLE II

CHEMICAL ANALYSES OF AS-RECEIVED POTASSIUM

FROM SHIPPING CONTAINER NO. 137

<u>Auxiliary Capsule No.</u>	<u>Zirconium Analyses, ppm</u>			<u>Potassium Impurities, ppm (Calculated)</u>		<u>Remarks</u>
	<u>O</u>	<u>N</u>	<u>H</u>	<u>O</u>	<u>N</u>	
3	1326	13	31.5	426	9	Potassium fill tube was not valved - capped in air
4	1229	27.5	39.5	406	5	Valves were placed at each end of potassium filled tube

NOTES: (1) Auxiliary capsule exposed in a vacuum at 1400°F for 100 hours.

(2) Cb-12Zr alloy reaction capsule, 1-inch diameter x 6-inch long.

(3) Initial analyses of zirconium, ppm

<u>Sample</u>	<u>O</u>	<u>N</u>	<u>H</u>	<u>C</u>
a	638	19	18	-
b	840	14	25	70
<u>c</u>	<u>587</u>	<u>31</u>	<u>40</u>	<u>135</u>
Avg.	688	21	28	103

(4) Auxiliary capsule #3 contained 8.9705 gm of potassium, 5.8035 gm of zirconium (20 square inch surface area).

(5) Analysis of oxygen, nitrogen and hydrogen in zirconium by vacuum fusion techniques.

(6) Potassium from shipping container No. 137 - slagged, filtered and hot trapped.

TABLE III

POTASSIUM ANALYSES BY THE ZIRCONIUM-GETTERING TECHNIQUE

<u>Auxiliary Capsule No.</u>	<u>Zirconium Analyses, ppm</u>			<u>Potassium Impurities, ppm (Calculated)</u>	<u>Remarks</u>
	<u>O</u>	<u>N</u>	<u>H</u>	<u>O</u>	
7	816	11	11	22	Potassium sampled during filling of AS-55 alloy capsules #11 and #12 and D-43 alloy capsules #2 and #4

- NOTES: (1) Auxiliary capsule exposed in a vacuum at 1400°F for 100 hours.
- (2) Cb-1Zr alloy reaction capsule, 1-inch diameter x 10-inch long x 0.080-inch thick wall.
- (3) Initial analyses of zirconium, ppm.

<u>Sample</u>	<u>O</u>	<u>N</u>	<u>H</u>	<u>C</u>
a	638	19	18	-
b	840	14	25	70
<u>c</u>	<u>587</u>	<u>31</u>	<u>40</u>	<u>135</u>
Avg.	688	21	28	103

- (4) Auxiliary capsule #7 contained 30.2 gm of potassium, 5.1427 gm of zirconium (20 square inch surface area).
- (5) Analysis of oxygen, nitrogen and hydrogen in zirconium by vacuum fusion techniques.
- (6) Potassium from shipping container No. 137 - slagged, filtered and hot trapped at MSA and re-hot trapped at General Electric for 200 hours at 1300°F.

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